NEW LABORATORY EXPERIMENTS IN PRACTICAL CHEMISTRY

NEWTON HENRY BLACK

TABLE OF THE MORE IMPORTANT ELEMENTS

with their symbols, approximate atomic weights, and valences

Element	Symbol	Approx. At. Wt.	Valence	Element	Symbol	Approx. At. Wt.	Valence
Aluminum	Al	27	III	Manganese	Mn	55	II IV
Barium	Ba	137	II	Mercury	Hg	200	I II
Bromine	Br	80	I	Nickel	Ni	58.7	II
Calcium	Ca	40	II	Nitrogen	N	14	III V
Carbon	C	12	IV	Oxygen	O	16	II
Chlorine	Cl	35.5	I	Phosphorus	P	31	III V
Chromium	Cr	52	II III VI	Platinum	Pt	195	IV
Copper	Cu	63.6	I II	Potassium	K	39	I
Fluorine	F	19	I	Radium	Ra	226	II
Gold	Au	197	I III	Silicon	Si	28	IV
Helium	He	4	——	Silver	Ag	108	I
Hydrogen	H	1	I	Sodium	Na	23	I
Iodine	I	127	I	Sulfur	S	32	II IV VI
Iron	Fe	56	II III	Tin	Sn	119	II IV
Lead	Pb	207	II IV	Tungsten	W	184	VI
Magnesium	Mg	24	II	Zinc	Zn	65	II

COMMON UNITS IN THE METRIC AND ENGLISH SYSTEMS

10 millimeters (mm.) = 1 centimeter (cm.)
100 centimeters = 1 meter (m.)
1000 cubic centimeters (cc.) = 1 liter (l.)
10 milligrams (mg.) = 1 centigram (cg.)
100 centigrams = 1 gram (g.)
1000 grams = 1 kilogram (kg.)

1 cc. of water at 4° C. weighs 1 gram
2.54 centimeters = 1 inch
1 meter = 39.37 inches
1 liter = 1.06 quarts
1 gram = 15.4 grains
28.35 grams = 1 ounce
1 kilogram = 2.20 pounds
1 pint of water weighs about 1 pound

GENERAL SUGGESTIONS

1. Follow Directions. Before performing an experiment, read the directions through carefully. Make sure that you understand the object of the experiment. Follow the directions closely; remember that every word is significant.

2. Notebook. A careful record of each experiment performed should be kept in a substantial notebook. Record the date, the title of the experiment, and the subheadings and paragraph letters (*e.g.* **a**, **b**).

Do not duplicate the printed directions. State briefly in your own words *what you did, what you observed,* and *what conclusions you drew.* Answer every direct question by a definite statement. Make a simple labeled diagram of the apparatus.

3. Quantities of materials. Do not take more of the materials than the amount specified. To take more means a waste of material and of time.

4. Disposal of waste. Such solids as matches, filter papers, bits of glass, and unconsumed chemicals should be thrown into the waste jars and not into the sink. If much acid or other liquid is emptied into the sink, flush it out with plenty of water.

5. Regulate your burner. The size of the burner flame should be regulated by the stopcock. A small flame is often better than a large one for heating test tubes and beakers.

6. Neatness is essential. Make sure your apparatus is clean. After each day's work clean up your apparatus and put your desk in order.

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A Chemical Laboratory in the Waltham (Mass.) High School.

NEW
LABORATORY EXPERIMENTS
IN
PRACTICAL CHEMISTRY

TO ACCOMPANY

BLACK AND CONANT'S
"NEW PRACTICAL CHEMISTRY"

BY

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PREFACE

This book has been arranged to accompany Black and Conant's *New Practical Chemistry.* The directions are framed primarily to meet the needs of students who are studying the subject for the first time. These experiments are the result of the author's long experience as a teacher of beginners in chemistry.

Laboratory work in any science is fundamental, and the study of a textbook merely extends and organizes the information that the student gets in the laboratory. The experiments which the student does with his own hands should make the fundamental principles of the science real and concrete to him. It is also essential for him to perform certain experiments which will indicate how these facts and principles are utilized at home and in the commercial and industrial life of the community.

The directions are at first very full and detailed, but they are gradually abbreviated in order to leave more and more to the student's own initiative and imagination. It is hoped that they are so clearly stated and illustrated that the teacher will be largely freed from the necessity of repeating the details of manipulation. Frequent questions (in italics) have been inserted in the directions to focus the attention of the student on the important facts to be observed.

One of the aims of all laboratory work is to arouse the student's enthusiasm for finding things out by experiment. To encourage this spirit, additional experiments, which are marked *Optional,* have been included. These may well be used to give elasticity and variety to the laboratory work as well as to emphasize the close connection between chemistry in the school laboratory and chemistry in the household, in the factory, or on the farm.

The *introductory paragraphs* are intended to give the setting of the experiment and to indicate its significance. These may well be amplified by the teacher in the oral discussion preceding each experiment.

It is expected that the instructor will select those experiments which meet the needs of his class. Probably *an average class of beginners in chemistry can in one school year perform successfully not more than forty of these experiments.* It is far better to have a small number carefully done, well written up, and thoroughly understood, than to rush through many carelessly.

Special care has been taken to select significant experiments and to word the directions so clearly that they can not be misunderstood. The author is greatly indebted to many teachers of chemistry for valuable suggestions. He wishes especially to mention Mr. M. E. Post, of the High School, Berkeley, California and Mr. Raymond S. Tobey, of the Girls' Latin School, Boston, Mass.

The apparatus required for the essential experiments is simple and the chemicals are inexpensive (see lists in Appendix). The *essential experiments* marked in the table of contents with a star (*) deal with fundamental facts and should be included in every first-year course.

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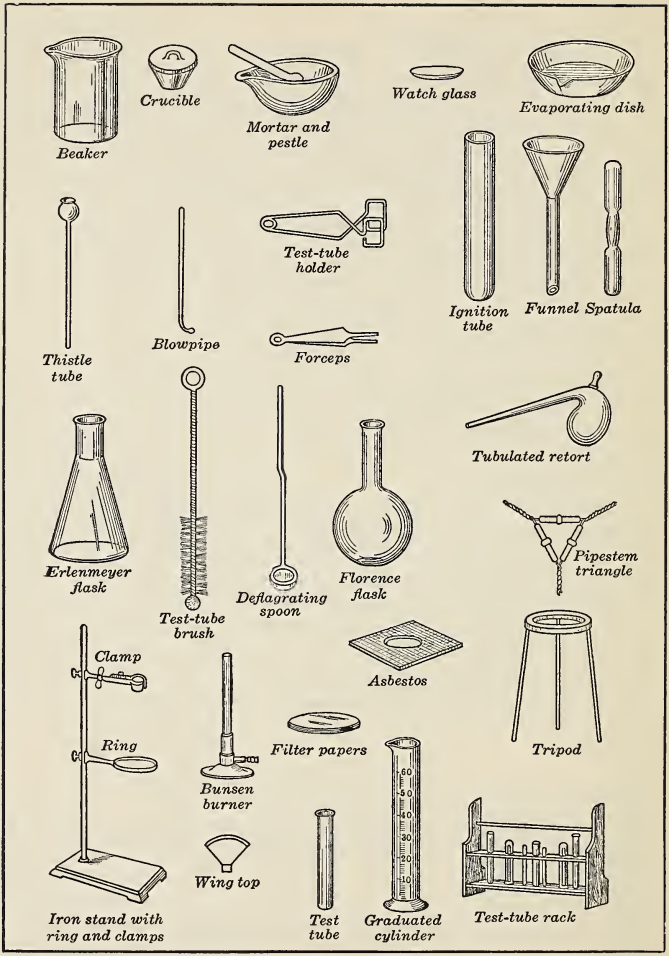
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Crucible
Mortar and pestle
Watch glass
Evaporating dish
Ignition tube
Funnel
Spatula
Thistle tube
Blowpipe
Test-tube holder
Forceps
Tubulated retort
Erlenmeyer flask
Test-tube brush
Deflagrating spoon
Florence flask
Pipestem triangle
Asbestos
Clamp
Ring
Bunsen burner
Filter papers
Tripod
60
50
40
30
20
10
Wing top
Test tube
Graduated cylinder
Test-tube rack
Iron stand with ring and clamps

NEW LABORATORY EXPERIMENTS IN PRACTICAL CHEMISTRY

HINTS ABOUT LABORATORY MANIPULATION

Check up your apparatus. Make sure that the outfit in your desk is complete and in good condition. On the opposite page you will find the names and pictures of some pieces of apparatus with which you must become familiar. Keep the articles clean and arranged in an orderly fashion.

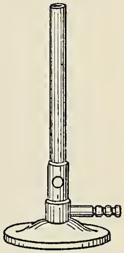

Fig. 1. Bunsen burner.

Bunsen burner. In a laboratory provided with gas the Bunsen burner (Fig. 1) is almost indispensable as a source of heat. When the air holes at the base of the burner are open, air is drawn in and mixes with the gas. It should burn with a clear, blue flame; the size of the flame can be controlled by adjusting the stopcock. When the holes at the base of the burner tube are closed, a yellow, luminous flame results.

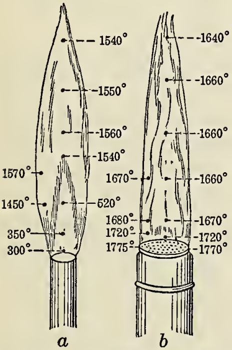

Fig. 2. Probable temperatures in (*a*) a Bunsen flame and (*b*) a Fisher flame.

In lighting such a burner, never hold the match over it while turning on the gas; turn on the gas and then apply the match. Otherwise it will probably "snap back," that is, burn at the base inside the tube. In this condition the burner gives off a disagreeable gas and becomes extremely hot. Turn off a "snapped" lamp, allow it to cool, and relight it properly.

In the **Fisher form of Méker burner** the flame is much hotter, as shown in figure 2. This is due to the fact that the gas is supplied with a large volume of air and that the grid on top of the burner prevents the flame from "striking back." In fact, there is practically a solid cone of burning gas.

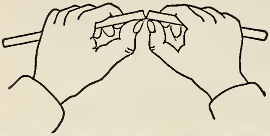
Fig. 3. How to break glass tubing which has been scratched.

Glass tubing. To cut a piece of small glass tubing, lay it on the table and make a scratch with a triangular file or glass-blower's knife at the point where it is to be cut. Next take the tube in both hands, placing the thumbs on the side opposite the scratch. Now press the thumbs against the tube and pull the ends toward you (Fig. 3).

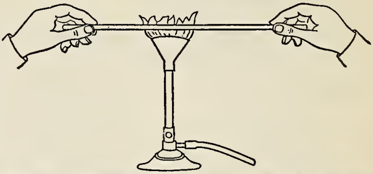
Fig. 4. Heating glass tubing in a flat flame.

To bend glass tubing, rotate it in a flat flame, such as that furnished by the **wing top** on the Bunsen burner (Fig. 4), until soft. Then take it out of the flame and gradually bend it into the desired shape. In order to make a good bend (Fig. 5), the tube must be heated evenly over a portion which is at least 5 cm. in length. Remember that hot glass placed on a cold table is almost sure to break.

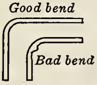

Fig. 5. A good and a bad bend.

Before glass tubes are used for any purpose, the sharp edges must be rounded by being heated near the top of a Bunsen flame. This is called "**fire-polishing**" the ends of the tube.

Setting up apparatus. In putting together apparatus it is often necessary to push a glass tube through a stopper. It is a good plan to lubricate the glass tubing with water and to insert the tube with a slow twisting motion — not by pushing. It is also very important to have the joints tight. To test apparatus for gas-tightness, either compress the air in it by blowing or draw some air out by suction. Since some part of the apparatus is generally under water, the level of the water can be made to rise or fall in a tube. If there is a leak, this level will change slowly when the opening where the lips were applied is closed. Do not try to plug up holes with matches or wax but *set up the apparatus properly.*

Heating apparatus. Wipe dry the outside of every glass or porcelain vessel before heating it. In heating a test tube containing a liquid, hold it in an inclined position with some kind of holder and keep it moving in the flame, taking care not to let the flame touch the glass above the liquid. **Don't try to boil a liquid in a flask or beaker with a direct flame.** Use the wire gauze with asbestos center. Turn the flame down until it is just the size of the beaker and boiling will begin sooner.

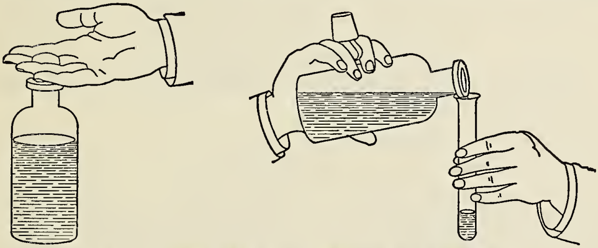

Fig. 6. Proper way to hold a stopper while pouring.

Porcelain dishes are used chiefly in evaporating and should be heated over asbestos. A porcelain crucible may be supported on the pipestem triangle and heated directly, provided it is warmed up gradually and cooled off slowly.

Handling materials. Don't try to *pour solids* out of a bottle. Use some kind of spoon or ladle. Don't weigh materials directly on the scale pans, use a piece of creased paper. Don't use more material than required and don't pour back excess material into the reagent bottle.

In pouring liquids from a bottle, some care must be taken to prevent the liquid from running down the side of the bottle.

Place the neck of the bottle lightly against the rim of the vessel into which the liquid is being poured. *The stopper must never be laid down on the desk.* Hold it between the fingers as shown in figure 6.

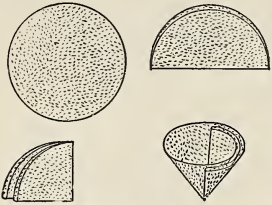

Fig. 7. Folding a filter paper.

To filter a liquid, first fold the filter paper in quarters and then open it up to form a cone (Fig. 7). Fit the cone into a funnel of such size that the paper does not quite reach the top and wet it thoroughly with water. The filter is now ready for use.

Stirring rods should be fire-polished. They are very convenient in pouring liquids. The liquid may easily be made to flow down the rod (Fig. 8). A stirring rod should also be used in testing with litmus paper. Put a drop of the liquid to be tested on the paper. Never dip the paper into the liquid.

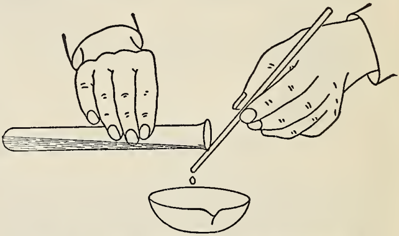

Fig. 8. Pouring a liquid down a glass rod.

Weighing and counting weights. In weighing out the materials for an experiment, use the **platform, or trip, scales** (Fig. 9). If these are in good condition, they are sensitive to one-tenth of a gram (0.1 g.). Remember that the thing to be weighed is put on the *left-hand* pan and the weights on the *right-hand* pan; remember to use the slider for weights under 10 grams.

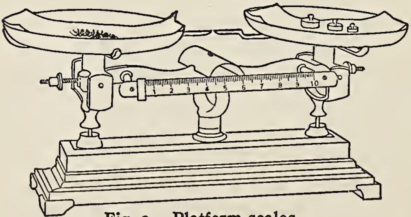

Fig. 9. Platform scales.

For **quantitative experiments** use the **horn-pan balance** (Fig. 10), which is sensitive to one-hundredth of a gram (0.01 g.).

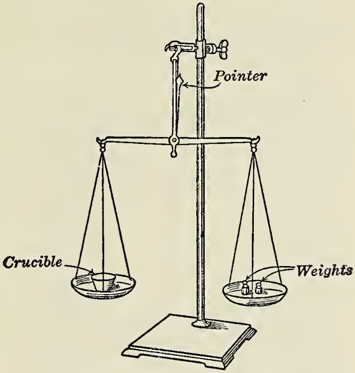

Fig. 10. Horn-pan balance.

A **triple-beam balance** (Fig. 11) is preferred by some because weighings can be quickly made from 0.01 up to 111 grams without the use of loose weights. An **analytical balance** (Fig. 12) is provided with agate bearings, and has a graduated beam with a 10-mg. rider; it is enclosed in a glass case. Such a balance should be sensitive to 0.1 mg. when loaded with

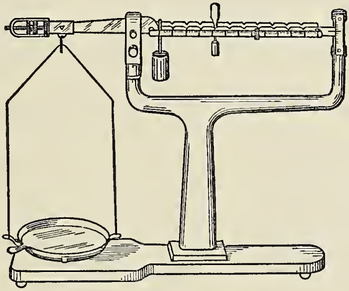

Fig. 11. A triple-beam balance with no loose weights.

100 grams. Examine the box of weights and see how they are arranged and marked (Fig. 13). Notice that the heavier weights (one gram and above) are made of brass and that the fractional weights are of German silver or aluminum and are marked in milligrams. Thus, 500 milligrams is 0.5 grams and 10 milligrams is 0.01 grams.

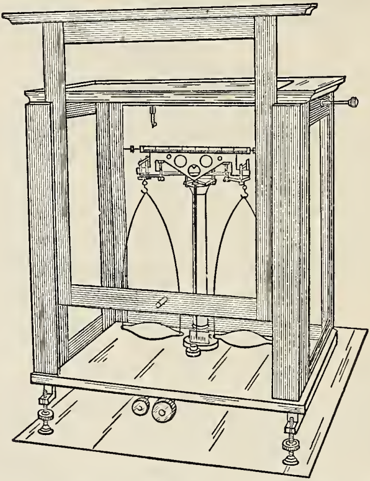
Fig. 12. An analytical balance with a graduated beam.

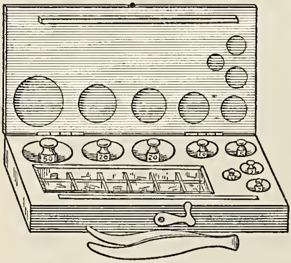

Fig. 13. Box of weights.

Use of the balance. In weighing, first see that your balance swings freely and evenly, that is, that the pointer swings an equal distance to the right and left. If it does not, adjust it either by means of a setscrew or by adding counterbalance. Place the object to be weighed on the left-hand pan and the weights on the right.

First try a weight which you think is a little heavier than the object; remove it if too heavy and try smaller ones in order. Never handle the weights with your fingers; always use the forceps. Count up the weights on the pan and write down the total at once in your notebook, not on a scrap of paper. *Experience shows that many mistakes in results are due to miscounting weights.* Check this result by noting those absent from the box. Beginning with the largest weight, return them to the box and again count them as you replace them. *In this way you double check yourself.*

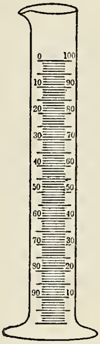

Fig. 14. Graduated glass cylinder.

Do not wait for a balance to come to rest. A good balance will continue to swing for a long time. Place just enough weights on the pan so that the pointer swings an equal distance to the right and left.

Measuring volumes. A graduated glass cylinder (Fig. 14) is commonly used to measure the volume of a liquid. Care must be taken to keep the eye on a level with the surface of the liquid. Get familiar with the scale markings and their value The eye should follow the scale from the zero end. It will also be noticed that the surface of water is not flat but curved. Such a curved surface is called a **meniscus.** With a concave meniscus, such as occurs on water, the position of the lowest point of the lowest curve against the scale is to be taken as the reading (Fig. 15). In measuring small volumes use a smaller graduated cylinder, a conical graduate, or a graduated test tube. For measuring rather large volumes of liquids we use *volumetric flasks* which are graduated on the neck to contain a definite volume at 20° C.

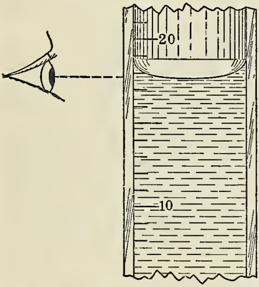

Fig. 15. Reading the meniscus of water (17.8).

HOW TO KEEP LABORATORY NOTES

It is a necessary part of the laboratory study of chemistry to keep in a notebook a neat and accurate record of all experimental work. It will be found helpful to follow the general order used in this manual with the same headings and subheadings. *Observations should be recorded immediately.* The author believes that the student should answer in complete sentence form all the italicized questions given in the directions. But these sentences should be concise.

The data obtained in quantitative experiments should be carefully labeled, and the result of each measurement should be placed on a separate line in tabular form. This is illustrated in the following table of sample data obtained for Exp. 6:

Wt. of crucible and potassium chlorate	22.03 g.
Wt. of crucible empty	20.30 g.
Wt. of potassium chlorate	1.73 g.
Wt. after first heating	21.42 g.
Wt. after last heating	21.35 g.
Wt. of oxygen (Loss of wt.)	0.68 g.
Percentage of oxygen in potassium chlorate	39 %

The data must be entered at once directly in the notebook, never on loose pieces of paper. All computations should also be made in the notebook. *Don't crowd your figures!*

It will save time in the end to acquire the habit of recording data legibly and of arranging the computations so that the method of calculation is evident at once. In general your data will be accurate only to *three significant figures* (p. **112**) and sometimes less, therefore you should keep only three significant figures in your calculations.

Do your experiments entirely independently. *Record what you have done and seen.* If you allow another student to perform the experiments for you or if you copy his notes, you are getting no benefit from this work, and you are wasting your own time.

HOW TO MAKE NOTEBOOK DRAWINGS

Don't try to make a picture of your apparatus, but make a **sectional drawing.** Imagine the apparatus to be cut by a vertical plane; suppose the sheet of paper to be placed in the position of this plane. Then draw the lines where the paper would cut the intersected apparatus. Figure 16 shows a sectional drawing of the apparatus used to make oxygen. It is also useful for review purposes to label the drawing. A carefully labeled diagram of your apparatus may well take the place of a word-description in your notes. Unless you are unusually skillful in free-hand drawing, *you had better use a ruler.*

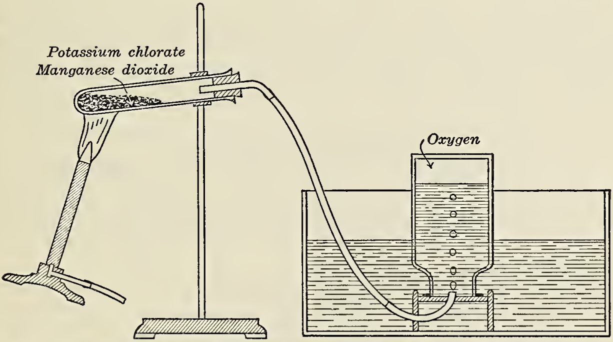

Fig. 16. Sectional drawing of oxygen-generating apparatus. (See also Fig. 27.)

EXPERIMENT A

Preliminary Experiment on the Metric System

Apparatus. — 30-cm. rule with inches on the reverse side. 100-cc. graduated cylinder. Test tubes (6″ × $\frac{3}{4}$″) and (8″ × 1″), small beaker, and evaporating dish. Horn-pan balance and set of weights.

Introduction. The metric system is based on the **meter** (m.), which is approximately 39.37 inches, or a little more than a yard. In the chemical laboratory we commonly use the **centimeter** (cm.), which is one-hundredth of a meter, just as the cent is one-hundredth of a dollar. It will be well to remember that 1 inch is equal to 2.54 centimeters or that 10 centimeters are approximately equal to 4 inches. This can be visualized by examining the scales in figure 17. In measuring very short distances, such as the diameter of a wire, we use a **millimeter** (mm.), which is one-thousandth of a meter, or one-tenth of a centimeter. In measuring longer distances out of doors, we use the **kilometer** (km.), which is one thousand meters, or about 0.62 miles.

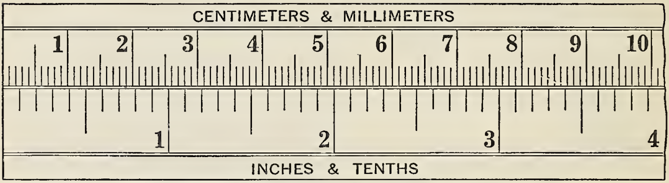

Fig. 17. English and metric units of length.

In measuring volumes, we commonly use the **cubic centimeter** (cc.), which is, of course, the volume of a cube one centimeter on each edge. For larger volumes we use the **liter** (l.), which is the volume of a cube 10 centimeters on each edge. Therefore a liter is equal to 1000 cubic centimeters. It is well to remember that a liter is about equal to one quart. One cubic centimeter is one-thousandth of a liter, hence the abbreviation, **ml.**, is sometimes used for the cubic centimeter.

In weighing objects in the chemical laboratory, we use the **gram** as the unit of weight. This is the weight of one cubic centimeter of water at 4° C., or 39° F. In elementary chemistry, we shall often need to weigh things to the nearest hundredth of a gram, or **centigram** (cg.). In analytical chemistry, we weigh to the nearest **milligram** (mg.), which is one-thousandth of a gram, and in atomic-weight determinations, we try to weigh to one-hundredth of a milligram, or one-hundred-thousandth of a gram. For larger weights, we use the **kilogram** (kg.), or 1000 grams, as the unit. It is well to remember that one kilogram is equal to 2.2 pounds.

SUMMARY OF IMPORTANT METRIC UNITS[*]

1 centimeter (cm.) = 10 millimeters (mm.)
1 meter (m.) = 100 centimeters (cm.)
1 kilometer (km.) = 1000 meters (m.)
1 inch = 2.54 centimeters
1 meter = 39.37 inches
1 liter (l.) = 1000 cubic centimeters (cc.)
1 liter = 1.06 quarts
1 gram (g.) = 100 centigrams (cg.)
1 kilogram (kg.) = 1000 grams
1 kilogram = 2.20 pounds

Directions. 1. *Measurement of length.* Examine the 30-cm. rule on both sides so as to become familiar with the relative sizes of the centimeter and the inch. Measure (*a*) the length of the page in this book, (*b*) the height of a test tube, and (*c*) the diameter of an evaporating dish (across the top). Measure first in centimeters and millimeters and record the result as a whole number of centimeters with a decimal to express the millimeters. Then express this measurement in inches and a decimal fraction (two places) of an inch. Use this relation: 2.54 cm. = 1 inch. *Record in your notebook your results in tabular form as follows:*

---

[*] These units should be memorized.

OBJECT	CENTIMETERS	INCHES
(*a*)	—— cm.	—— in.
(*b*)	—— cm.	—— in.
(*c*)	—— cm.	—— in.

2. *Measurement of volume.* Measure the volume of (*a*) your large test tube by nearly filling it with water and then pouring the water carefully into your cylindrical graduate. In reading the surface of the water in the graduated cylinder, be sure your eye is on a level with the surface to be measured. Also read the position of the bottom of the *meniscus*, that is, the concave, or crescent-shaped, surface of the water. You should also read to the nearest marked division (cc.) on the glass cylinder and then estimate to one-tenth of a division. Then you will record in your tabular form the volume in cubic centimeters and tenths of a cubic centimeter. In the same way, measure the volume of (*b*) a small beaker and (*c*) your evaporating dish (when nearly full). *Make in your notebook a table somewhat like the following:*

OBJECT	CUBIC CENTIMETERS
(*a*)	—— cc.
(*b*)	—— cc.
(*c*)	—— cc.

3. *Measurement of weight.* Weigh an empty beaker on your **horn-pan balance** (Fig. 10). This balance should be supported so that the pans are about 2 inches above the table. Such a balance, even with a load of 100 grams, is sensitive to 0.01 gram. Place the beaker on the *left* scale pan and then

with the forceps place on the *right* scale pan a weight which you think is a bit too heavy to balance the beaker. If it proves to be heavier, replace it with the next smaller weight. If this is too light, add to the right scale pan the next smaller weight. In this manner proceed to add weights until you have the beaker balanced so nearly that one centigram (0.01 g.) can be detected in the movement of the pointer of the balance as it swings back and forth. Remember that a good balance should not quickly come to rest!

Now fill the beaker about half full of water and weigh the beaker and water. Then compute the weight of the water in the beaker. Also pour all the water into your graduated cylinder and find its volume. In this way you can check the accuracy of your weighing because one gram of water occupies one cubic centimeter. *Why do you not expect the volume in cubic centimeters to check exactly with the weight in grams? It will be convenient to record in your notebook your weights in tabular form:*

Object	Weight
Beaker + Water	—— g.
Beaker empty	—— g.
Weight of Water	—— g.
Volume of Water	—— cc.

EXPERIMENT B

Preliminary Experiment in Laboratory Manipulations

Apparatus. — Bunsen burner. Rubber tubing. Triangular file. Wing top.

Material. — Asbestos paper (6″ × 6″). Glass tubing (about 30 cm. long and 7 mm. in diameter). Pins. Matches.

Introduction. We shall use the type of gas burner now almost universally found in chemical laboratories. It was invented in 1855 by a famous German chemist, Robert Wilhelm Bunsen (1811–1899), who taught for many years at the University of Heidelberg. If coal gas is burned in an ordinary jet, the flame is smoky. But when the gas is mixed with a suitable amount of air, the flame is much hotter and does not deposit soot.

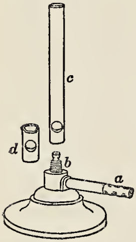

Fig. 18. Parts of Bunsen burner.

Caution. A flame with a "roaring" sound means that too much air is entering the burner. If the flame "strikes back" and burns noisily at the base of the burner, it means that there is not enough gas pressure and too much air. Turn off the gas and start again. But be sure to give the burner a chance to cool off before you touch it.

1. Bunsen burner. Identify the essential parts of the burner, such as the gas orifice, collar, air holes, and barrel, shown in figure 18. Attach it by means of the rubber tubing to the gas pipe. Turn on the gas. (a) *What is the difference in the character of the flame when the air holes are open and when they are closed?* (b) *What is the effect of regulating the supply of gas at the stopcock?*

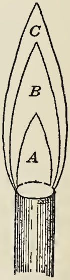

Fig. 19. Parts of the Bunsen flame.

2. Bunsen flame. If we look at the flame when the holes are open, we shall see that there are two cones, an inner cone *A* which is blue and an outer one *B* which is bluish-purple

(Fig. 19). The space A within the inner cone is filled with a mixture of gas and air. This mixture is partially burned in B and completely burned in C. We may show that the space A contains unburned gas and is cool:

(*a*) We may place a match on a pin and suspend it on the top of a Bunsen burner as shown in figure 20. When we turn on the gas and light it, *why does the match head not take fire at once?*

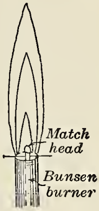

Fig. 20. Match inside the Bunsen flame.

(*b*) We may hold a sheet of asbestos paper vertically against the flame as shown in figure 21, and then quickly remove it. Repeat with the paper held horizontally. We shall find that the paper has been scorched. *What does the inner space within the charred area indicate?*

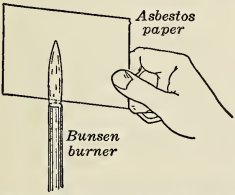

Fig. 21. Scorching paper in a Bunsen flame.

3. Handling glass tubing. (*a*) To cut a piece of glass tubing, we first scratch the tube at the point where we wish to cut it. This may be done with a triangular file. Then we hold the tube in both hands with the thumb nails opposite the scratch. By bending the tubing and at the same time pulling the hands apart, the tube will break clean across. (*b*) To bend glass tubing, we first place the wing top on the Bunsen burner and light the burner and adjust the collar to give a blue flame. Hold a 6-inch piece of glass tubing in the flame so that about 2 inches in the middle will be heated. Rotate the tube slowly in your fingers so that it will be heated evenly on all sides. As soon as the tube where heated has become quite soft, remove the tube from the flame and slowly bend it until the two arms are at right angles. *Why may the tube be red hot in the middle and yet can be easily handled at the ends?* To test the bend, lay it on an asbestos square. It should lie flat. Also the bend should be rounded and not sharply angular as shown in figure 5. After the tube has become cool enough to handle in the middle, the ends should be **fire-polished.** To do this we hold the end in the upper part of the ordinary blue Bunsen flame. When the flame turns yellow, it means that the glass has begun to soften and the sharp edges at the end are rounded off. *Why should the ends of a glass tube be fire-polished?*

EXPERIMENT 1

Changes in Metals Heated in Air

Apparatus. — Forceps. Bunsen burner. Tripod and pipe-stem triangle. Lid of porcelain crucible. Iron wire 6″ long.

Materials. — Copper wire (# 24). Magnesium ribbon (6 cm.). Tin (mossy). Lead strip (2 cm. × 0.5 cm.). Aluminum wire (# 18).

Introduction. When wood or coal burns, it seems to disappear except for the ashes left behind. About one hundred and fifty years ago chemists began to study this process. Their discoveries enabled the science of chemistry to advance rapidly. The true explanation of burning was found by studying the change which many metals undergo when heated in air. In this experiment we shall observe how certain metals change when heated.

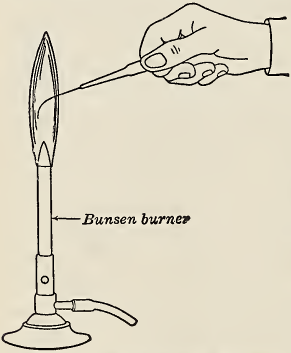

Fig. 22 Heating wire in Bunsen flame.

Directions. a. *Copper.* Examine a piece of bright copper wire and record those characteristic properties which enable one to recognize it as copper, such as its *color, hardness, luster,* and *heaviness.* Hold the wire with forceps in the tip of the flame of a Bunsen burner (Fig. 22) until you are sure that longer heating will produce no further change.

Cool and compare the surface material with a piece that has not been heated.

How has the copper changed?

NOTE. Always state the experimental evidence on which you base your conclusions.

You might well record your observations in this experiment in a tabular form like the following:

METAL HEATED	PROPERTIES	
	BEFORE HEATING	AFTER HEATING
Copper		
Magnesium . . .		
Lead		
Aluminum . . .		
Tin		

b. ***Magnesium.*** Repeat the experiment (a), using a piece of magnesium ribbon. *Record definite properties of magnesium and of the product, such as color, appearance (metallic or otherwise), and pliability or brittleness.*

c. ***Lead.*** Repeat the experiment (a), using a strip of lead. First scrape it in order to get a fresh metallic surface.

d. ***Aluminum.*** Repeat the experiment (a), using a piece of aluminum wire.

e. ***Tin.*** Place the lid of a crucible on a pipe-stem triangle which is supported on a tripod. On this lid heat a few pieces of granulated tin. Always apply heat to a porcelain vessel gently at first by waving the flame about under the vessel. Then turn down the flame and set the burner under the lid. Stir the melted tin with an iron wire. *Compare the original tin with the product.*

EXPERIMENT 2

Change in Weight of a Metal Heated in Air

Apparatus. — Porcelain crucible. Horn-pan balance and weights. Ring stand. Tripod and pipe-stem triangle. Bunsen burner.
Materials. — Fine steel wool (# 00).* Carbon tetrachloride.

Caution. Never place chemicals of any sort directly upon the scale pans. Never weigh an object while it is hot. Why not?

Directions. a. Weigh a clean, dry porcelain crucible (without cover) to the nearest centigram (0.01 g.). Place steel wool in the crucible and weigh the whole as before. Support the crucible on a pipe-stem triangle (Fig. 23) and heat, at first gently and then with the full Bunsen flame, for 15 minutes. The flame should be adjusted so that the crucible is just above the tip of the inner blue cone. For the purposes of this experiment it is not necessary that the action be complete.

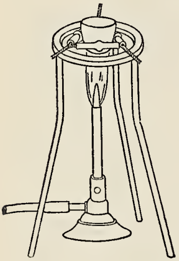

Fig. 23. Heating a crucible on a pipe-stem triangle.

b. To cool the crucible, gradually lower the flame and finally remove it, allowing the crucible to cool on the triangle until it can be held in the hand.

Weigh the crucible again.

Has there been a loss or a gain in weight? To what is the difference in weight probably due?

Record your weights in tabular form as follows:

Wt. of crucible + iron . . . . g.
Wt. of crucible empty . . . . g.
    Wt. of iron before heating . . . . g.
Wt. of crucible + iron after heating . . . . g.
Wt. of crucible empty . . . . g.
    Wt. of iron after heating . . . . g.
    Change in wt. of iron . . . . g.

---

* The steel wool may have a trace of oil on it. This should be removed by washing the steel wool with carbon tetrachloride and drying it.

EXPERIMENT 3

Separating a Mixture into Its Components

Apparatus. — Test tubes and corks to fit. Funnel. Filter papers. Crystallizing dish. Small flask. Evaporating dish. Ring stand.
Materials. — Black gunpowder. Carbon bisulfide.

Introduction. A material as found in nature is rarely a pure substance. It consists of at least two substances more or less uniformly distributed throughout the material and loosely or firmly held together as the case may be. Such a material is called a **mixture,** and its ingredients are called **components.** Each component retains its identity and its own set of physical and chemical properties. But the properties of a mixture are intermediate between the properties of its components. In this experiment we shall show that black gunpowder, although apparently homogeneous, is really a mixture of three quite different substances: sulfur, saltpeter, and charcoal. The problem, then, is to make use of certain properties of each substance to separate it from the mixture.

Directions. a. Examine some black gunpowder. Although it is a mixture of three different substances, yet they are so carefully put together that they are not easily discernible. However, we can separate the various components by using suitable solvents. Carbon bisulfide will dissolve the sulfur, and water will dissolve the niter, or saltpeter; and the charcoal, which is insoluble in both of these solvents, is left behind.

b. Put about 5 g. of gunpowder into a test tube to which a cork has been fitted. Pour upon the gunpowder 10 cc. of carbon bisulfide, replace the cork, and shake the test tube for 5 minutes. Allow it to settle, pour off the clear liquid through a dry filter (Fig. 24) into a beaker or crystallizing dish, and set it aside where no flame will get at it.

Caution. Carbon bisulfide is very inflammable and must never be handled near a flame.

The carbon bisulfide will soon evaporate and leave crystals of sulfur.

c. Spread the black residue left in the test tube on a filter paper to dry. When it has dried, transfer the residue to a small flask. Pour 20 cc. of hot water upon it and shake for 5 minutes. The saltpeter has dissolved, and the solution may be filtered into a clean evaporating dish. By heating the filtrate, the water is evaporated. A white residue, the **saltpeter**, or **niter**, is left.

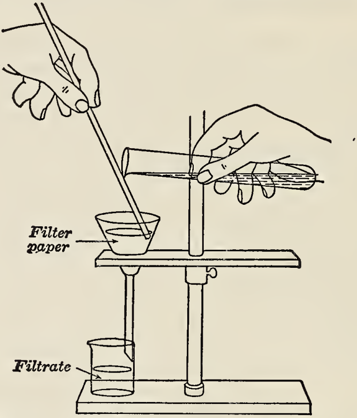

Fig. 24. Separating a mixture by filtration.

d. The black residue left on the filter paper is **charcoal**, or carbon, which is the third component of gunpowder.

Results. *What property of each of the three components of gunpowder was made use of in this separation?*

EXPERIMENT 4

Decomposition of a Compound

Apparatus. — Ignition test tube (Pyrex) with 1-hole rubber stopper. Ring stand and clamp. Pneumatic trough. Test tubes. Glass tubing. Bunsen burner. Wing top.

Materials. — Mercuric oxide. Wood splint.

Introduction. It was Lavoisier who discovered the nature of the substance which is taken from the air when a metal is heated. He heated mercury for several days, keeping the temperature a little below its boiling point, and obtained a small amount of red powder. Then he heated this powder in a glass tube to a temperature above the boiling point of mercury. To be sure, this experiment had been done some time before by Priestley, but he did not understand the true significance of his results.

In this experiment we shall take some of this same red powder, heat it, and find the properties of the gas produced.

Directions. **a.** Place about 2 or 3 g. of the red powder (mercuric oxide) in the bottom of a clean ignition tube (Pyrex test tube). This can be done by putting the powder near the end of a narrow strip of folded paper and sliding it carefully into the tube, as shown in figure 25. By inclining the tube and gently tapping the paper, the powder will be deposited in the bottom of the tube. This method leaves the sides of the tube perfectly clean.

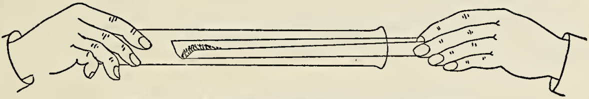

Fig. 25. Sliding a powder into a test tube.

Now fit the ignition tube with a one-hole rubber stopper carrying a delivery tube which is arranged as shown in figure 26. Fill a test tube with water and invert it over the end of the delivery tube in a dish of water (pneumatic trough).

b. Warm the ignition tube very gradually by holding the burner in the hand and waving the flame along the tube. Very soon bubbles of gas will be seen coming from the end of the delivery tube.

What is the cause of this bubbling?

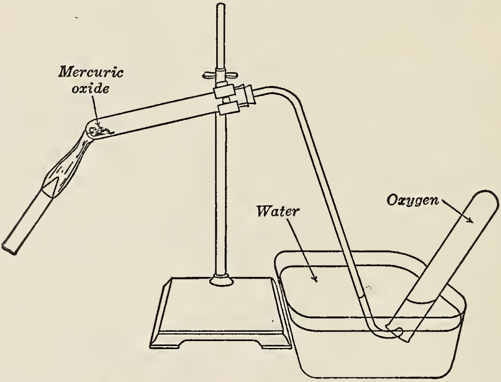

Fig. 26. Decomposing mercuric oxide.

When half a test tube of gas has been collected, remove the test tube, invert it, and test the gas by thrusting into it a glowing (not flaming) splint. *Is the gas air?*

Caution. Never allow a hot ignition tube to cool while it is connected with a delivery tube which has its free end under water.

Explain what would happen.

c. Continue heating the ignition tube until all the powder is decomposed. Collect a second test tube of gas and test with a glowing splint. This gas is oxygen. *What is the difference in the behavior of the glowing splint in the two test tubes?*

d. Scrape off with a splint some of the substance left on the side of the ignition tube. *What is it?*

Into what two substances has the red powder been decomposed?

EXPERIMENT 5

Oxygen — Preparation and Properties

Apparatus. — Test tube and 1-hole stopper. Ring stand with clamp. Bunsen burner. 4 wide-mouth bottles. 4 glass squares. Pneumatic trough. Deflagrating spoon. Glass tubing.

Materials. — Potassium (or sodium) chlorate. Manganese dioxide. Wood splint. Splinter of charcoal. Copper wire (15 cm. # 18). Sulfur. Red phosphorus. Asbestos paper.

Introduction. The mercuric oxide used in experiment 4 is too expensive to use in large quantities to produce oxygen. In this experiment we shall therefore use potassium chlorate, a substance containing much oxygen. A black powder, manganese dioxide, will also be added. The latter will, when the experiment is completed, be left in the tube unchanged. It has, however, the remarkable property of enabling the potassium chlorate to give off its oxygen at a lower temperature and more evenly. Such a substance which changes the speed of a chemical reaction without being itself permanently altered is called a **catalytic agent.**

Preparation. Mix on paper about 10 g. of potassium chlorate and about one-third its bulk of manganese dioxide. Slide the mixture into a test tube, clamp it horizontally, and tap it so as to distribute the mixture along the side of the lower half of the tube, leaving space above the mixture to the end of the tube. Insert the rubber stopper carrying the delivery tube as shown in figure 27. Fill the pneumatic trough with enough water to cover the support for the bottles. Fill 4 wide-mouth bottles with water, cover them with glass plates, and invert them in the trough.

Use a small Bunsen flame to heat the tube, keep the flame moving, and remove the flame when the gas comes too fast. Heat slowly. *Will the gas which passes over at first be pure oxygen?* Now fill the bottles with oxygen one by one, covering each when full with a glass plate and setting it mouth upwards on the table. After the fourth bottle is filled, remove the delivery tube from the water and allow the test tube to cool. The residue in the test tube may easily be removed with warm water.

Properties. a. Use one bottle of the gas to determine such properties as color, odor, and solubility, and then thrust in a glowing splint. (Sometimes a slight cloud is present when oxygen is made from potassium chlorate, because of the presence of an impurity.) The solubility can be inferred from the method of collecting the gas.

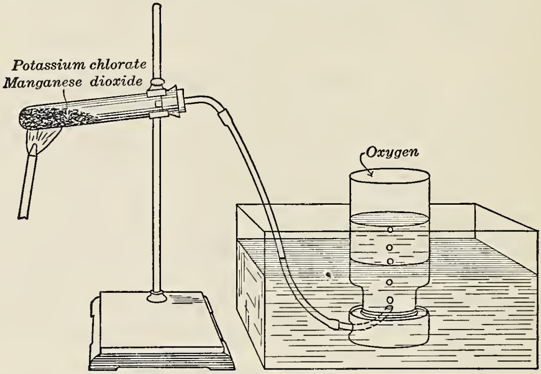

Fig. 27. Decomposing potassium chlorate.

b. *Charcoal.* Wind one end of a piece of wire around a thin splinter of charcoal. Heat the charcoal until it glows brightly and immediately lower it into a bottle of oxygen. *Compare the combustion of charcoal in oxygen with that in air. Of what does the gas produced probably consist?*

c. *Sulfur.* Line a combustion, or **deflagrating, spoon** with asbestos paper and put into it about 0.5 cc. of sulfur. Ignite the sulfur in the Bunsen flame. Observe the flame for a moment and then lower the spoon into a bottle of oxygen, keeping the mouth of the bottle closed as far as possible with the glass plate. *Compare the flame of sulfur burning in oxygen with that* *in air. Is it finally extinguished? If so, why?* Very cautiously smell the contents left in the bottle. *What is it?*

d. *Phosphorus.* Clean and reline the spoon with asbestos. Place in it a bit of red phosphorus (size of small pea), ignite and lower it into another bottle of oxygen. *Compare the burning as before. Is the product a gas or does it consist of fine solid particles?*

Caution. Before putting the spoon away, clean it by heating it to redness for two minutes to burn up all the phosphorus.

Results. *Record the properties of oxygen which you have observed in this experiment. What is its most striking characteristic?*

EXPERIMENT 6

Percentage of Oxygen in Potassium Chlorate

(Quantitative Experiment)

Apparatus. — Porcelain crucible and cover. Ring stand. Horn-pan balance. Set of weights. Pipe-stem triangle. Iron forceps. Bunsen burner.

Materials. — Dry potassium chlorate.

Introduction. Since potassium chlorate is a chemical compound, it will be interesting to determine its percentage of oxygen. All chemical compounds have a definite percentage composition. Later we shall see how it is possible to determine the percentage of chlorine and potassium in the potassium chlorate. Since this is a quantitative experiment, it will be well to read again at this point the special directions concerning weighing, counting of weights, and the use of balances.

Directions. Clean and dry a porcelain crucible and cover. Weigh the empty crucible to a centigram (0.01 g.). Put about 2 grams of *dry* powdered potassium chlorate into the crucible and weigh again as before.

Place the covered crucible on the pipe-stem triangle and support it on the ring stand at some convenient height (Fig. 28). Begin heating with a small Bunsen flame (about 5 cm. high) very gently. Lift the cover occasionally with the forceps and adjust the flame so that the melted potassium chlorate gives off its oxygen slowly. If some should spatter upon the cover, withdraw the flame and remove the cover to a clean piece of smooth paper. Loosen the bits of solid matter with a pin and very carefully return them to the crucible. Continue heating until the bubbling slackens, then heat strongly for 10 minutes. Now lower the flame to permit the crucible gradually to cool and finally remove it. When it is cold, weigh it again to the nearest centigram (0.01 g.).

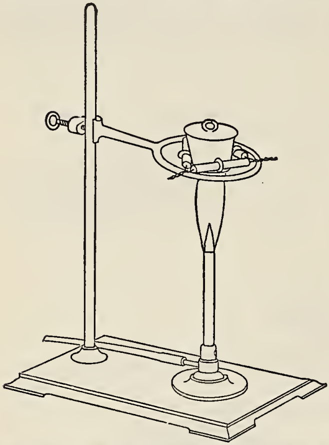
Fig. 28. **Heating potassium chlorate in a covered crucible.**

Again heat very hot for about 5 minutes, cool, and weigh as before. If the weight has not changed during the second heating, we may consider that the oxygen has been completely expelled. If the weight is less, heat once again for 5 minutes and continue in this way until two successive weights are the same. This process is called **heating to constant weight.**

The loss of weight is equal to the weight of the oxygen. The residue in the crucible is called **potassium chloride.**

The percentage of oxygen in potassium chlorate may be calculated thus:

$$\frac{x}{100} = \frac{\text{Wt. of oxygen}}{\text{Wt. of potassium chlorate}}$$

Make all computations in your notebook. Arrange your work in tabular form:

Wt. of crucible + potassium chlorate . . . . g.
Wt. of crucible empty . . . . g.
Wt. of potassium chlorate . . . . g.
Wt. after first heating . . . . g.
Wt. after last heating . . . . g.
Wt. of oxygen . . . . g.
Percentage of oxygen in potassium chlorate . . . . %

Compute the average of the percentages found by all in the class who have done this experiment. *What law of chemistry is illustrated?*

EXPERIMENT 7

Hydrogen — Preparation and Properties

Apparatus. — Wide-mouth bottle with 2-hole stopper. Thistle tube. Pneumatic trough. Glass tubing. 3 wide-mouth bottles and glass squares. Bunsen burner. Test tube.

Materials. — Zinc (mossy). Dilute sulfuric acid (1:6). Copper sulfate solution. Wax tapers.

Preparation. A convenient method of preparing hydrogen is furnished by the action of zinc on dilute sulfuric acid. Place about 20 grams of zinc in a bottle provided with a two-hole rubber stopper carrying a thistle tube and a delivery tube. The apparatus is arranged for collecting the gas over water (Fig. 29).

Insert the stopper tightly with a twisting motion and pour through the thistle tube enough dilute sulfuric acid to cover the zinc. *Why should the thistle tube dip into the acid?* If the gas does not come off freely, add through the thistle tube a few drops of copper sul-

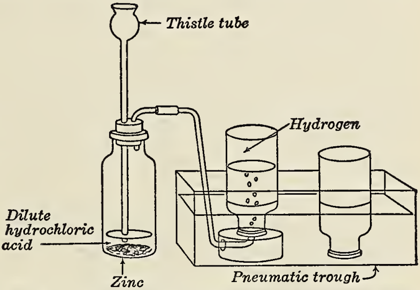

Fig. 29. Generating hydrogen by the action of zinc on dilute sulfuric acid.

fate solution. But do not add more acid to the generator without consulting the instructor.

The gas which passes over first is evidently a mixture of hydrogen and air. A confined mixture of hydrogen and air, if ignited, explodes with great violence. To determine when the hydrogen is free of air, collect a test tube full of the gas, carry it mouth downward to a small flame, and ignite the gas. Continue to collect and test the gas in this way until a portion does not give a sharp explosion but burns quietly. Then collect 3 bottles of hydrogen, cover each with a glass plate, and set mouth downward on the desk.

Properties. **a.** Allow one bottle of hydrogen to stand uncovered (mouth up) for a minute. Then bring a lighted taper to the mouth of the bottle. *What does the result show about the density of hydrogen as compared with air?*

b. Light a short taper and lift a second bottle of hydrogen (mouth downward). Insert the burning taper almost to the bottom of the bottle and then withdraw it slowly. *What happens to the flame of the taper? What occurs at the mouth of the bottle? Explain.*

c. Place a bottle of hydrogen, mouth downward, over a bottle of air, mouth upward. Allow the bottles to remain mouth to mouth in this position for 3 minutes. Carry first the lower bottle and then the upper bottle quickly to a flame. *What fact about the diffusion of gases does this experiment illustrate?*

d. Filter the liquid remaining in the flask and evaporate to about one-half its volume in a porcelain dish over asbestos. Allow the liquid to stand until crystals (zinc sulfate) form. These crystals are composed of zinc, sulfur, and oxygen. *Complete in your notebook the equation for the reaction:*

zinc + sulfuric acid ⟶ ------------ + ------------

e. *Summarize the properties of hydrogen and compare with those of oxygen.*

EXPERIMENT 8

Reduction of an Oxide with Hydrogen

Apparatus. — Wide-mouth bottle with 2-hole stopper. Thistle tube. Drying tube with stopper. Ring stand and clamp. Bunsen burner. Test tube. Glass tubing.

Materials. — Copper oxide (wire form). Zinc. Dilute sulfuric acid. Absorbent cotton. Fused calcium chloride.

Introduction. We have already seen that bright metallic copper turns to black copper oxide when heated in the air (Exp. 1). In this experiment we shall reverse the process of oxidation and change copper oxide back to metallic copper; this is called **reduction.**

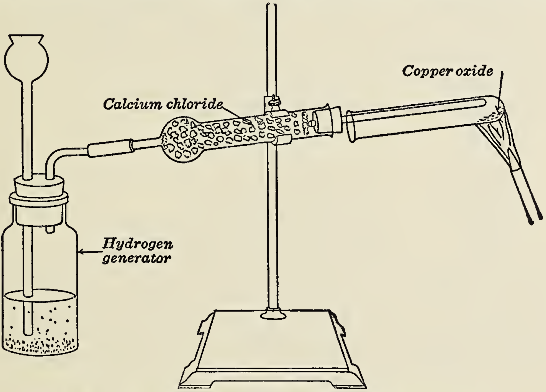

Fig. 30. Reducing copper oxide with dry hydrogen.

For this purpose we shall use dry hydrogen (free from water vapor), which can be obtained by passing the gas from the generator through a tube filled with granulated calcium chloride. This drying material is held in position by loose plugs of cotton placed at each end of the tube.

Directions. a. Place about 0.5 cc. of black copper oxide in the bottom of a dry test tube and place the latter in a slightly inclined position with mouth downward (Fig. 30). Insert a straight piece of glass tubing into the stopper of the drying tube and introduce this tubing into the test tube until the end touches the copper oxide.

b. Now generate hydrogen as in Experiment 7, and wait 3 minutes to allow all the air to be replaced by hydrogen.

Caution. Don't bring a flame near the apparatus until all the air is driven out.

Cautiously heat the copper oxide to redness, being careful to keep the flame away from the mouth of the tube.

c. *Note any change in the copper oxide. What collects in the cool part of the tube? What substance is reduced and what substance is oxidized in this experiment?*

Why is it necessary to dry the hydrogen?

EXPERIMENT 9

Electrolysis of Water*

Apparatus. — Hoffman electrolysis apparatus (Fig. 31). Battery jar (4″ × 5″). Two test tubes. Ring stand with two clamps. Two copper insulated wires with platinum electrodes attached. Storage battery (3 cells) or 4 dry cells (new) or a 100-watt lamp and socket with connections for 110-volt direct current.

Materials. — Water solution of sulfuric acid (1 vol. acid to 15 vol. water). Wood splints.

Introduction. Although this experiment may be done as a class demonstration, there is a genuine thrill which the beginner will experience if he performs it for himself. The object of the experiment is not only to show that water is a combination of oxygen and hydrogen, but also to determine the relative volumes of each gas. Since pure water is a very poor conductor of electricity, we shall add a very little sulfuric acid to increase its conductivity. But if you were to test the quantity of acid left in the water after you had been using the apparatus, you would find the quantity of sulfuric acid unchanged.

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* Unless the laboratory is equipped with electricity at each table, this experiment should be done as a CLASS EXPERIMENT by the instructor.

Directions. (*Class experiment.*) Set up the Hoffman electrolysis apparatus, if it is available, as shown in figure 31. A 6-volt storage battery or 4 new dry cells in series or, better, a 110-volt direct current with a 100-watt lamp in series may be used as a source of electricity. Fill the reservoir nearly full of water to which has been added one volume of sulfuric acid to 15 volumes of water.

Caution. To prepare dilute sulfuric acid always pour the acid slowly into the water — never the reverse.

Connect the battery with the electrode terminals. Bubbles of gas collect at each electrode and rise in the liquid. The electrode connected with the positive terminal of the battery is called the *anode* and that connected with the negative terminal is the *cathode*. *Compare the volumes of gases collected in the two tubes.* When the volume of gas at the cathode has reached 50 cc., disconnect the wires and measure the volumes in the tubes carefully.

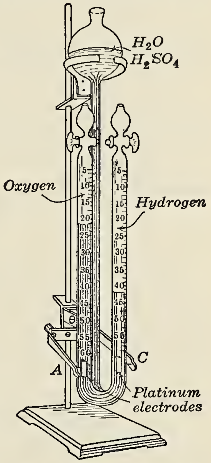

Fig. 31. Hoffman apparatus for the electrolysis of water.

To test the gas in the cathode tube, open the stopcock at the top and apply a lighted splint. *What happens?* Then apply a glowing splint to the stopcock at the top of the other tube. *What happens?*

Note. The tubes above the stopcocks should be dried out carefully with filter paper before applying the above tests, otherwise the sputtering drops of acid will interfere.

(*Student experiment.*) **a.** Fill a small battery jar about two-thirds full of water containing about one part by volume of sulfuric acid to 15 parts of water. Fill two test tubes with the dilute sulfuric acid, cover the end of each tube with your thumb, and invert it into the water of the jar. Slip each tube into its clamp and push it down until the platinum electrode is entirely within the tube as shown in figure 32. Each electrode consists of a strip of platinum foil soldered to a piece of rubber-covered copper wire. The exposed copper wire and soldered joint should be covered with paraffin or sealing wax. Wash your hands thoroughly to remove every trace of the acid.

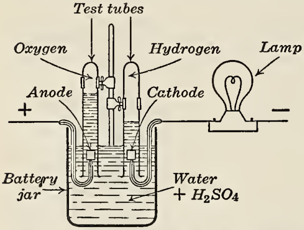

Fig. 32. Electrolysis of water with student's form of apparatus.

b. Connect the two electrodes with a 6-volt storage battery, or with a 110-volt direct-current circuit having a 100-watt lamp in series. Note the positive (+) and negative (−) terminals. We shall assume in this experiment that electricity enters the solution at the + electrode (anode) and leaves the solution at the − electrode (cathode).

c. As soon as the instructor has inspected your apparatus, turn on the current. *How does the volume of gas liberated at the anode compare with the volume liberated at the cathode during the same length of time?*

When the water in one of the tubes has been displaced by the gas, remove the tube from the battery jar. Holding the mouth of the tube downward, bring it near a flame. *What is the gas?*

As soon as the other tube is filled with gas, close it with your thumb and remove it from the jar. Invert the tube and insert a glowing splint. *What is the gas? What gas is collected at the cathode? What gas is collected at the anode?*

Assume that there is the same weight of sulfuric acid in the battery jar at the end of the experiment as at the beginning. *Where did the gases come from? Complete the word-equation:*

water ⟶ ------------- + -------------

What was the purpose of adding the sulfuric acid?

EXPERIMENT 10

Distillation of Water

Apparatus. — Liebig condenser. Boiling flask. Flask with 1-hole stopper. Ring stand. Wire gauze with asbestos center. Bunsen burner. Wide-mouth bottle. Test tubes. Glass tubing.

Materials. — Common salt (sodium chloride). Potassium permanganate (or copper sulfate). Ammonia water (concentrated). Litmus paper (red). Marble chips or glass beads.

Introduction. Natural water is never absolutely pure. To make it fit for drinking and for industrial purposes, filtration, boiling, or treatment with a chemical, such as chlorine, is resorted to. But for laboratory use it is absolutely essential to distill the water. This process consists in boiling the water and condensing the steam. If the apparatus is clean and if the impurities have a higher boiling point than the water, the distillate is pure. The following experiment illustrates the process.

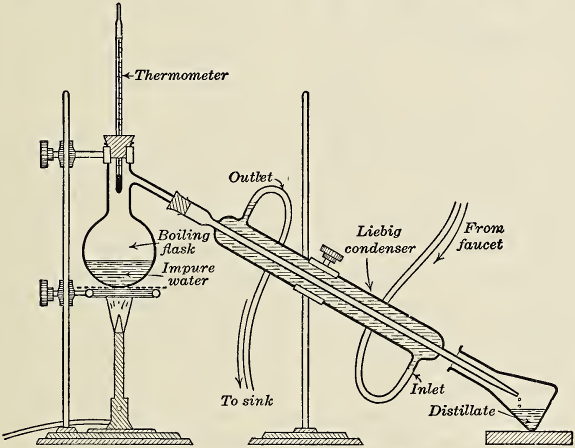

Fig. 33. Distillation of water with Liebig condenser.

Directions. (*Class experiment.*) a. Set up the Liebig condenser and boiling flask as shown in figure 33. Use about 100 cc.

of ordinary water and add to it one drop of concentrated ammonium hydroxide solution. Add enough copper sulfate to give it a distinctly blue color. In each of six clean test tubes place one drop of phenolphthalein solution (an indicator which turns pink with ammonia) and catch the distillate in these test tubes. Change the test tube as soon as 15 cc. of liquid has come over.

Questions. *Does the ammonia pass over with the steam? Does the copper sulfate pass over with the steam? Do the first portions of the distillate contain more or less ammonia than the later portions?*

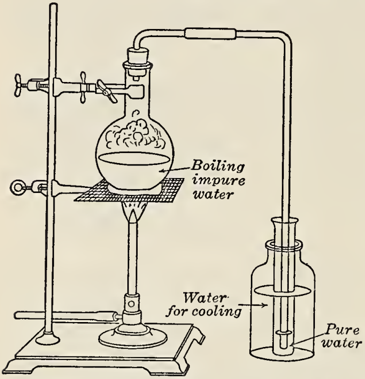

Fig. 34. Simple apparatus for distilling water.

(*Student experiment.*) b. Dissolve a spoonful of common salt in about 50 cc. of water. Put the solution in a flask which is arranged as in figure 34. A few chips of marble in the flask will prevent "bumping." The steam from the boiling water in the flask is condensed by conducting it into the test tube, which is kept cold by the water in the bottle. Boil gently until 5 cc. has distilled over. Taste the distilled water. *Is it free from salt? Explain.*

Repeat, using 50 cc. of water colored with a crystal of potassium permanganate. *Is the distillate colorless?* (Don't taste it.)

c. Add a few drops of concentrated solution of ammonia (a gas) to 50 cc. of water. Note the odor of the liquid and place a drop of it on red litmus paper. *Result?*

Distill the dilute ammonia water in the same way in which you did the salt water. *What is the odor of the distilled liquid?* Test it with red litmus paper. *Is it free from ammonia? Explain.*

EXPERIMENT 11

Measurement of Water of Hydration

(Quantitative Experiment)

Apparatus. — Ring stand. Pipe-stem triangle. Porcelain crucible. Horn-pan balance and weights. Bunsen burner.

Materials. — Barium chloride (or gypsum or copper sulfate) crystals.

Introduction. The amount of water of crystallization contained in a hydrate, such as gypsum, copper sulfate, or barium chloride, is a definite quantity and varies with different crystallized substances. It can be determined by heating a known quantity of the crystallized substance until all of the water is driven off. We can tell when the process is complete because then the substance ceases to lose weight on further heating. We use barium chloride because it is easily obtained pure.

Directions. a. Weigh a clean, dry crucible with cover to the nearest centigram (0.01 g.). Place about 3 grams of crystallized barium chloride (do not try to take an exact amount) in the crucible and weigh again with cover carefully. Record these weights in tabular form as shown below.

b. Place the covered crucible on the pipe-stem triangle and place this on the ring stand, as shown in figure 28. Heat the crucible at first very gently, holding the burner in the hand. *Why begin heating gently?* Then place the burner under the crucible and adjust the flame so that the top of the inner blue cone is a short distance below the crucible. After heating for 10 minutes, remove the cover and continue heating strongly 5 minutes longer. Then *gradually* cool the crucible and reweigh to the nearest centigram.

c. Reheat the crucible (without cover) for 5 minutes, allow it to cool, and reweigh. Repeat this operation until the weight is constant. *Why? From your results calculate the percentage of water of hydration present in the crystals.*

Wt. of crucible + barium chloride crystals . . . . g.
Wt. of crucible empty . . . . g.
Wt. of barium chloride taken . . . . g.
Wt. after first heating . . . . g.
Wt. after last heating . . . . g.
Wt. of water of hydration . . . . g.

$$\text{Percentage of water} = \frac{\text{wt. of water}}{\text{wt. of barium chloride}} \times 100 = \quad . \ . \ . \quad \%$$

EXPERIMENT 12

Replacement of Hydrogen by a Metal

(Quantitative Experiment)

Apparatus. — Gas-measuring tube, 50-cc. Battery jar (about 4″ × 5″). Horn-pan balance and weights. Thermometer. Barometer.
Material. — Magnesium ribbon. Conc. hydrochloric acid. Thread.

Introduction. In this experiment we shall determine the weight of magnesium required to generate 1 gram of hydrogen. This is called the **equivalent weight** of magnesium. We shall need (1) to take a known weight of magnesium, (2) to displace hydrogen from an acid with this quantity of the metal, (3) to measure the volume of hydrogen replaced, and (4) knowing the density of hydrogen (grams per liter), to compute the weight of this hydrogen. (5) Finally, we may calculate from the weight of magnesium used and the weight of the hydrogen evolved the weight of magnesium needed to give one gram of hydrogen; thus,

$$\frac{x}{1} = \frac{\text{Wt. of magnesium used}}{\text{Wt. of hydrogen evolved}}$$

Directions. It will be well to fill a small battery jar about $\frac{3}{4}$ full of water at the temperature of the room.

a. Since magnesium ribbon is very nearly uniform in size, its weight may be considered proportional to its length. We may then measure off several meters of clean, bright ribbon (enough for the whole class) and weigh it as precisely as possible with the best balances available. From these data we can compute the weight of 1 cm. of ribbon and the length of magnesium ribbon which will weigh about 0.045 grams. *Record the weight of magnesium actually used.*

b. Roll the magnesium ribbon into a loose coil but small enough to slide easily into the gas-measuring tube. Attach a piece of thread to the coil. Pour about 5 cc. of concentrated hydrochloric acid into the gas-measuring tube and then fill it with water slowly in order to avoid as much as possible mixing the acid and water.

c. Now put the weighed piece of magnesium ribbon into the measuring tube and lower it about 5 cm., holding it by the thread so that it will not sink too far. Then place your thumb over the mouth of the tube and invert it into the battery jar of water. Let the tube rest on the bottom of the jar so that it holds the thread, as shown in figure 35.

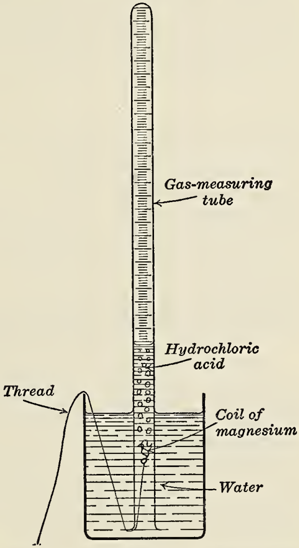

Fig. 35. Apparatus for determining the equivalent of magnesium.

Since the acid is heavier (sp. gr. 1.19) than water, it will gradually flow downward in the tube and react with the magnesium. The magnesium may be allowed to rise nearly to the graduated portion of the tube. If any magnesium sticks to the sides of the glass tube as the liquid goes down, rinse it off by shaking the glass. *What is the gas which is collected? From what substance does the gas come?* Copy and complete the following word-equation:

magnesium + *hydrochloric acid* ⟶ ---------- + ----------

d. When the action has ceased and the liquid is clear of bubbles of gas, raise or lower the measuring tube so that the surface of the liquid inside the tube is at the same level as the surface of the liquid in the jar. Then *read very carefully the volume of the hydrogen collected* and record it in your notebook in a table similar to the one shown below.

TABLE

Data:

Weight of magnesium used . . . . g.
Volume of hydrogen obtained . . . . cc.
Temperature of water in jar . . . . ° C.
Barometric pressure . . . . mm.
Density of hydrogen under these conditions . . . . g./cc.

Computations:

Weight of the hydrogen evolved . . . . g.
Weight of magnesium needed to get 1 g. hydrogen . . . . g.

Since the density of hydrogen depends on the temperature and pressure, it will be necessary to *record also the temperature of the liquid in the jar* and *the barometric pressure.*

From the table in the Appendix giving the density of hydrogen at various temperatures and pressures, *find the density of hydrogen* (grams per liter) *at the temperature and pressure in this experiment. Record in your notebook the density of hydrogen as collected,* expressed as grams per cubic centimeter.

From this density as given in the table and the volume you have measured, *calculate the weight of the hydrogen collected.*

Finally, *calculate the weight of magnesium needed to get 1 gram of hydrogen.*

EXPERIMENT 13

Analysis of an Unknown Mixture *

(Quantitative Experiment)

Apparatus. — Ignition test tube (Pyrex), 6″ × $\frac{3}{4}$″. Ring stand and clamp. Bunsen burner or Fisher form of Méker burner. Horn-pan balance and weights. Porcelain crucible.

Materials. — Unknown mixture of potassium chloride and potassium chlorate (furnished by your instructor). Shredded asbestos.

Introduction. In this experiment you will have an opportunity to apply your chemical formulas as well as your skill in weighing. The problem is to determine the composition of a mixture of potassium chloride and potassium chlorate by finding the loss of weight in the sample due to the evolution of oxygen upon heating. An unknown sample (numbered) will be furnished by the instructor.

Directions. Clean and dry an ignition test tube and fasten a loop of copper wire around it near the open end. Suspend the tube from the hook over the pan of your horn-pan balance and weigh to the nearest centigram (0.01 g.). Place about 2 grams of the unknown sample in the test tube. Be sure that none of the powder sticks to the walls of the tube. Now weigh the tube again with the sample in order to determine the weight of the sample within a centigram.

Prepare two plugs of shredded asbestos by first heating the asbestos in a porcelain crucible in order that it may be thoroughly dry. Push one plug, about 15 mm. long, down into the ignition tube until it is about 5 cm. from the bottom of the tube. Push the second plug into the tube until it is about 9 cm. from the bottom. Again weigh the loaded ignition tube as carefully as before.

Now clamp the ignition tube so that it is nearly horizontal but so that the closed end is a little lower than the open end.

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* This experiment is based on a similar one used in the laboratories of Chemistry A at Harvard University.

Heat the tube, at first gently, until the contents begin to melt. Keep up this slow decomposition for about 5 minutes. Then heat the residue more vigorously for 3 minutes.

Caution. Do not heat the tube to such a point that white fumes appear above the second asbestos plug.

Allow the tube to cool to the temperature of the room and reweigh as precisely as your balance allows.

Calculations. *How much oxygen was evolved from your sample? How much potassium chlorate was needed to produce this oxygen? What percentage of your mixture was potassium chlorate?*

EXPERIMENT 14

Decomposition of Water by Sodium

Apparatus. — Knife. Evaporating dish. Forceps. Ring stand and clamp. Pneumatic trough. Large test tube. Glass plate. Stirring rod.

Materials. — Sodium metal. Litmus-paper strips, red and blue. Lead foil.

Caution. The reaction of sodium with water is violent. Don't handle sodium with wet hands. Follow the directions.

Introduction. Water is the most familiar of all chemical compounds. We have already seen [p. 22] * that it may be decomposed by an electric current into two entirely different substances — oxygen and hydrogen. Hydrogen can be got from water, even at ordinary temperatures, by means of certain active metals such as sodium and potassium. But it is necessary to follow directions carefully since these metals react vigorously with the water. However, for laboratory purposes we use a much cheaper and less active metal such as zinc; and because the action of zinc on water is too slow, we add an acid like dilute sulfuric or hydrochloric acid. (See Experiment 7.) A great many metals have the power to drive hydrogen out of

* Bracketed numbers refer to pages in Black and Conant's *New Practical Chemistry.*

an acid solution and to take its place in the solution. In recent years electricity has become so cheap a form of energy that much of the hydrogen which is used commercially is prepared by the electrolysis of water.

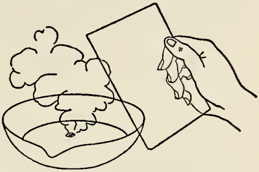
Fig. 36. Violent action of sodium on water.

Directions. a. What is the appearance of a freshly cut surface of sodium? *Is sodium hard or soft? Heavy or light?*

b. Fill an evaporating dish half full of water, and float upon it a piece of filter paper. Then with the forceps drop on the filter paper a bit of sodium (size of the head of a match). It is well to protect the eyes against spattering drops with a plate of glass (Fig. 36). *Observe the results.*

The filter paper keeps the sodium in place, thus preventing it from spinning around on the surface of the water.

c. After the action has ceased, wet your fingers with the solution and rub them together. *Result?* Test with a stirring rod the action of a drop of the solution on red and on blue litmus paper. *Result?*

d. Wrap a clean, freshly cut piece of sodium (about 3 mm. in diameter) in dry lead foil (tea lead). Punch several holes in the lead with a knife blade. Holding the lead in forceps, quickly slip it under the mouth of a large inverted test tube filled with water (Fig. 37). If necessary, use another piece of sodium wrapped in lead to complete the filling of the tube. If the sodium escapes from the lead covering, stand back!

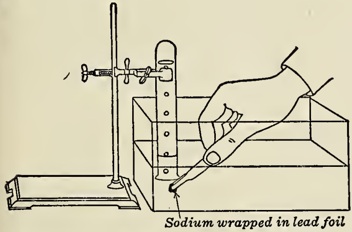

Fig. 37. Collecting the gas evolved from sodium and water.

When the tube is filled with gas, carry it mouth downward to a flame and ignite the gas. *Result?* This gas is hydrogen, and the yellow flame is due to sodium. *If sodium is an element, where does the gas come from?*

Draw a labeled diagram of the apparatus used in (**d**).

Copy in your notebook and complete the word equation:

Water + sodium ⟶ sodium hydroxide + --------

EXPERIMENT 15

Chlorine — Preparation and Properties

(*Class Experiment*)

Apparatus. — Flask with 2-hole stopper. Ring stand with large ring and clamp. Thistle tube. Bunsen burner. Pan or water bath. 4 wide-mouth bottles and glass plates. Pneumatic trough. Mortar and pestle. One hydrogen generator for class. Glass tubing.

Materials. — Manganese dioxide. Concentrated hydrochloric acid. Powdered antimony. Taper.

Introduction. Next to water, probably the most familiar compound is common salt, which we shall show to be made up of the metal sodium and the gas chlorine. Commercially chlorine is prepared by the electrolysis of salt water; but in the laboratory it is more convenient to make it from concentrated hydrochloric acid. This acid is treated with an oxidizing agent which oxidizes the hydrogen of the acid to water and sets free the chlorine. In this experiment we shall use manganese dioxide (MnO_2) as the oxidizing agent. The reaction is as follows:

$$\underset{\text{hydrochloric acid}}{4\,HCl} + \underset{\text{manganese dioxide}}{MnO_2} \longrightarrow \underset{\text{manganese chloride}}{MnCl_2} + \underset{\text{water}}{2\,H_2O} + \underset{\text{chlorine}}{Cl_2\uparrow}$$

In this reaction the manganese unites with one-half of the chlorine of the hydrochloric acid; the rest of the chlorine is evolved as a gas when the mixture is warmed.

Caution. **Chlorine is a poisonous gas. Avoid inhaling much chlorine. Breathing ammonia gives some relief. This experiment should be done under a hood or in a place where there is a good draft.**

Preparation. Set up the apparatus as in figure 38. Place about 10 g. of granular manganese dioxide in the generating flask and pour in about 25 cc. of concentrated hydrochloric acid. Rotate the flask in order to mix the contents thoroughly.

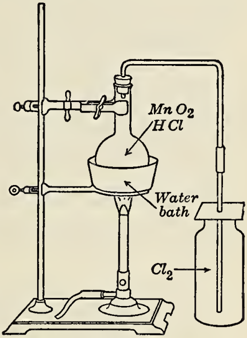

Fig. 38. Preparation of chlorine.

Heat the water in the dish under the flask to boiling. Fill 4 wide-mouth bottles by the displacement of the air, holding a piece of white paper behind the bottles to see when they are full. *Note the color of the gas.* Cover the bottles with glass plates and set them aside.

Properties. a. *Solubility in water.* Slide one of the glass plates aside and fill one bottle of chlorine about one-third full of cold water. Cover it with the palm of your hand and shake the contents vigorously. Lower the bottle into a trough of water and remove your hand. *What can you say about the solubility of chlorine?*

b. *Action with metals.* Grind up in a mortar a fragment of antimony and sprinkle some of the powdered antimony (Sb) into a bottle of chlorine. *Name the product* ($SbCl_3$) *and write the equation. Is oxygen always necessary for combustion?*

c. *Action with hydrogen.* Introduce a jet of burning hydrogen into a bottle of chlorine. Blow gently across the mouth of the bottle and note the change of color. *Name the product and write the equation for the burning.*

d. *Action with hydrocarbons.* Lower a lighted taper or candle into a bottle of the gas by means of a wire handle. Blow gently across the mouth of the bottle. *Name two products of this action.*

e. *Summarize the properties of chlorine which you have observed in this experiment.*

EXPERIMENT 16

Hydrochloric Acid

Apparatus. — Ring stand with clamp and large ring. Bunsen burner. Asbestos gauze. Florence flask with 2-hole stopper. Thistle tube. Wide-mouth bottles and glass squares. Glass tubing. Cardboard. Dish. Stirring rod.

Materials. — Sodium chloride (common salt). Concentrated sulfuric acid. Litmus papers. Concentrated ammonium hydroxide. Wood splint. Filter paper. Magnesium.

Introduction. Of the acids, undoubtedly sulfuric (oil of vitriol) is the most important. In this experiment we shall find out what happens when it is added to common salt. The gas which is evolved on heating, known as hydrogen chloride, has an extremely pungent odor, and therefore it is advisable to do this experiment in a well-ventilated place (or under a hood). The water solution of hydrogen chloride is called hydrochloric acid.

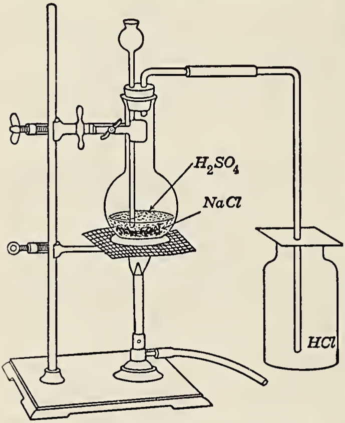

Fig. 39. Preparing hydrogen chloride.

Preparation. Arrange a flask with stopper, thistle tube, and delivery tube as shown in figure 39. Pour into the flask 7 cc. of water and then slowly add 20 cc. of concentrated sulfuric acid (H_2SO_4).

Caution. Always pour the sulfuric acid into the water.

Cool the diluted acid by holding the flask in a stream of running water. When the acid is cold, put into it about 15 g. of common salt (NaCl). Replace the stopper carrying the thistle tube and delivery tube and set the flask on the asbestos gauze. Clamp the neck of the flask at the right height for heating with a Bunsen burner. The thistle tube must extend almost to the bottom of the flask so that its end may be under the liquid. *Why?* Put the delivery tube through a piece of cardboard and let it extend nearly to the bottom of a clean, dry bottle.

Heat the flask very slowly with a small flame. You can tell when the bottle is full of the hydrogen chloride gas by holding a strip of moist blue litmus paper at the mouth of the bottle. When the paper turns red, the bottle is full of the gas (HCl) and should be replaced at once by another. Collect three bottles of gas and cover them with glass plates. *Complete in your notebook the equation:*

$$\underset{\substack{\text{sodium}\\ \text{chloride}}}{\mathrm{NaCl}} + \underset{\substack{\text{sulfuric}\\ \text{acid}}}{\mathrm{H_2SO_4}} \longrightarrow \underset{\substack{\text{sodium}\\ \text{hydrogen}\\ \text{sulfate}}}{\mathrm{NaHSO_4}} + \text{------}$$

Then fill another bottle about one-third full of distilled water and place the end of the delivery tube just below the surface of the water. Continue heating and observe the currents in the water at the end of the delivery tube. *What causes them?* Avoid letting the hydrogen chloride gas escape into the room.

Properties. **a.** Invert a bottle of the gas in a dish of water, remove the glass plate, and *note what happens. What does this show?*

b. Test the gas in another bottle with a lighted splint. *Is it combustible? Is it a supporter of combustion?*

c. Pour a little ammonium hydroxide on a wad of filter paper and throw it into a bottle of hydrogen chloride. *What is the result? Complete in your notebook the equation:*

$$\underset{\text{ammonia}}{\mathrm{NH_3}} + \underset{\substack{\text{hydrogen}\\ \text{chloride}}}{\mathrm{HCl}} \longrightarrow \text{------}$$

d. Hydrochloric acid is the water solution of hydrogen chloride. Dip a stirring rod into the solution and apply a drop to red and blue litmus papers.

Pour some of the hydrochloric acid just made into a test tube and drop into it a strip of magnesium. Test the gas set free with a flame. *Result? Complete in your notebook the equation:*

$$Mg + \_\_ HCl \longrightarrow \_\_\_\_\_\_ + \_\_\_\_\_\_$$

EXPERIMENT 17

Test for a Chloride

Apparatus. — Test tubes and rack.

Materials. — Dilute hydrochloric acid. Dilute nitric acid. Solutions of silver nitrate (5%), ammonium hydroxide, disodium phosphate, and potassium oxalate. "Unknown" solution.

Directions. **a.** Fill a test tube half full of dilute **hydrochloric acid** and add a few drops of silver nitrate solution ($AgNO_3$). The solid which separates out, *i.e.* the **precipitate,** is silver chloride ($AgCl$). *Complete the equation in your notebook:*

$$HCl + AgNO_3 \longrightarrow \_\_\_\_\_\_ + \_\_\_\_\_\_$$

Record the color and appearance of this precipitate. Label and set the tube aside for further experimenting.

b. In the same way add a few drops of silver nitrate solution to a solution of **disodium phosphate.** The precipitate in this case is silver phosphate.

Record its color and appearance. Label the test tube and set it aside.

c. To a solution of **potassium oxalate** add a few drops of silver nitrate solution. The precipitate is silver oxalate.

Record its color and appearance. Label this test tube and set it aside.

d. Try the effect of dilute **nitric acid** on each of the three precipitates just prepared. *How can you distinguish between silver chloride, silver phosphate, and silver oxalate?*

e. Prepare another portion of silver chloride by adding silver nitrate solution to a solution of **sodium chloride** (common salt). *Complete the equation in your notebook:*

$$NaCl + AgNO_3 \longrightarrow \_\_\_\_\_\_ + \_\_\_\_\_\_$$

Find out whether this precipitate (AgCl) is soluble in ammonium hydroxide.

f. Prepare still another portion of silver chloride and stand the tube in the **sunlight.** *What is the result?*

Summarize the three characteristic properties of silver chloride. How would you test for a soluble chloride?

g. Apply these tests to an "**unknown solution**" which you may obtain from the instructor. In making your report on this unknown solution, record the result of each step in the process of testing as well as your final conclusion. That is, *report the reaction of successive additions of silver nitrate, ammonium hydroxide, and nitric acid and your decision as to whether it is a chloride.*

EXPERIMENT 18

Hypochlorous Acid — Bleaching

(*Optional Experiment*)

Apparatus. — Test tubes and rack. Carbon dioxide generator (Kipp). Funnel.

Materials. — Bleaching powder (chloride of lime). Filter papers. Marble chips. Washing soda. Sodium sulfite. Concentrated hydrochloric acid. Colored calico. Litmus paper.

Preparation. Make a saturated solution of bleaching powder (chloride of lime) by adding about 2 cc. of the powder to 20 cc. of water in a test tube and by shaking occasionally. Filter off the clear solution and pass carbon dioxide (made by the action of hydrochloric acid on marble) through the liquid for at least 5 minutes. The equation for this reaction is as follows:

$$\underset{\text{chloride of lime}}{CaOCl_2} + \underset{\text{carbonic acid}}{H_2CO_3} \longrightarrow \underset{\text{calcium carbonate}}{CaCO_3\downarrow} + \underset{\text{hypochlorous acid}}{HOCl} + \underset{\text{hydrochloric acid}}{HCl}$$

By filtering off the precipitate, calcium carbonate, we obtain a clear solution containing the hypochlorous acid.

Use in bleaching. Place in the solution of the hypochlorous acid, just prepared, small strips of (1) litmus paper, (2) paper with printing on it, (3) paper on which there is writing in ink, (4) paper with pencil marks, and (5) colored calico. *Record the effect on each. Which of these materials is colored with organic compounds and which with free carbon? Does hypochlorous acid oxidize free carbon? What does it oxidize?*

Bleaching. Unbleached cotton cloth may be bleached with chloride of lime as follows: (1) First clean the cloth of grease by boiling it in a weak solution of washing soda (5% Na_2CO_3 solution). (2) Then wash and place the cloth in a bleach bath (10% $CaOCl_2$ solution). (3) After 5 minutes partially wring the cloth and place it in a very dilute solution of sulfuric acid (5 cc. conc. H_2SO_4 in 100 cc. water). (4) Again place the cloth in the bleach solution and thus alternate until the color is removed. (5) To remove any traces of the hypochlorous acid left in the cloth, wash it several times in a solution of sodium sulfite (5% Na_2SO_3 sol.). Mount in your notebook a sample of the original cloth and another as bleached.

EXPERIMENT 19

Acids, Bases, and Salts

Apparatus. — Test tubes and rack. Stirring rod. Evaporating dish. Tripod and asbestos gauze. Glass plate. Bunsen burner.

Materials. — Dilute hydrochloric, sulfuric, nitric, and acetic acids. Litmus papers (red and blue). Solutions of sodium hydroxide, potassium hydroxide, ammonium hydroxide, and calcium hydroxide. Sodium hydroxide (sticks or pellets).

Directions. **a.** *Acids.* Prepare dilute solutions of the following acids by adding a few drops of each to 10 cc. of water: hydrochloric, sulfuric, nitric, and acetic. Lay a piece of blue litmus paper and a piece of red litmus paper on a glass plate. Touch both kinds of paper with the end of a clean glass rod wet with each acid in turn. *What changes do you observe?*

Taste *one* drop of each of the dilute solutions and immediately rinse the mouth with water. *What is the distinctive taste of acid?*

Write the formulas of these acids (see Chap. XII) *and find out what element is present in all of them.*

b. *Bases.* In a similar way test with litmus papers dilute solutions of the following bases: sodium hydroxide, potassium hydroxide, ammonium hydroxide, and calcium hydroxide. *What is the characteristic change in litmus which you observe with bases?*

Taste a drop of calcium hydroxide solution. *How would you characterize the taste of bases?*

Write out the formulas of these bases and find out in what respect they are similar in composition.

c. *Salts.* Dissolve a small piece of sodium hydroxide (a stick about 1 cm. long or 6 pellets) in about 20 cc. of water in a test tube. Stir it until all is dissolved and then pour out about 15 cc. of the solution into an evaporating dish (Fig. 40). Add dilute hydrochloric acid a little at a time and keep stirring the liquid until a drop on a glass rod gives no basic reaction with litmus.

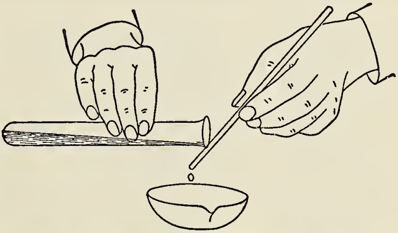

Fig. 40. Neutralizing a base with an acid.

Then dilute the 5 cc. of sodium hydroxide solution which was left in the test tube until it is five times as dilute, and also dilute the acid solution the same amount. Now use these much diluted acid and base solutions to bring the solution in the evaporating dish to the neutral point, or **end point,** *i.e.* the point where it affects neither red nor blue litmus paper. This process is called **neutralization.**

Evaporate the neutral solution to dryness slowly so that the liquid does not spatter. Taste the residue. *What is it?*

The scientific name for the residue is sodium chloride, which indicates that it contains only sodium and chlorine. *What has probably become of the other elements in the acid and base?*

Complete the equation in your notebook:

$$NaOH + HCl \longrightarrow NaCl + \_\_\_\_$$

EXPERIMENT 20

Concentration of a Solution by Titration

(Quantitative Experiment)

Apparatus. — Two burettes. Small Erlenmeyer flask (50 cc.). Stirring rod. Ring stand with 2 clamps.

Materials. — Fifth-normal solution of sodium hydroxide (8 g. to 1 liter) and a solution of hydrochloric acid (nearly the same concentration, made by diluting 34 cc. of conc. hydrochloric acid to 1 liter).* Phenolphthalein solution (1 g. dissolved in 100 cc. of 50% alcohol).

Introduction. **A normal solution of a base** contains 17 g. of replaceable hydroxyl (OH) per liter. *How many grams of sodium hydroxide (NaOH) must be dissolved in water and made up to a liter to form a fifth-normal (0.2 N) solution?*

A **normal solution of an acid** contains 1 g. of replaceable hydrogen per liter. *How many grams of hydrogen chloride (HCl) does one liter of a normal hydrochloric acid solution contain?*

Directions. a. Fill one burette nearly full of a sodium hydroxide solution of known concentration (0.2 N). Label the burette and clamp it in a vertical position. Then fill another burette with the hydrochloric acid, the concentration of which is to be determined (Fig. 41). Draw from each burette enough liquid to remove the air bubbles from the tip and to bring the **meniscus** (the curved surface of the liquid) down to the graduated portion of the burette.

---

* The precise amount of material needed to make one liter of normal solution (NaOH or HCl) can be purchased in a small glass-stoppered bottle under the trade name "Fixanal."

b. Examine carefully the markings on the burettes in order to understand the graduations. In reading a burette, make sure that the eye is on a level with the lowest part of the meniscus, as shown in figure 15. *Record the readings of both burettes in tabular form as indicated on page 52.*

NOTE. Observe that the graduation is downwards. If then the reading is four-tenths of a cc. above the 1 cc. mark, the reading is 0.6 cc. (not 1.4 cc.).

c. Let about 10 cc. of the basic solution flow out into a little flask and then add a drop or two of some indicator, such as **phenolphthalein solution.** Remember that this indicator turns a pink color when the solution is basic; an acid solution remains colorless.

d. Now set the flask on a sheet of white paper under the other burette and allow the acid to run in a few drops at a time. Shake the solution in the flask vigorously after each addition of a few drops. When the acid begins to decolorize the indicator near the point where it enters, work more cautiously. Add one drop at a time and shake the solution until the pink color just disappears. Now add a drop or two of the base until the reddish tinge just appears; see whether one drop of acid will make the solution colorless. Continue in this way until a reversal of color is got by *one drop* of base or acid.

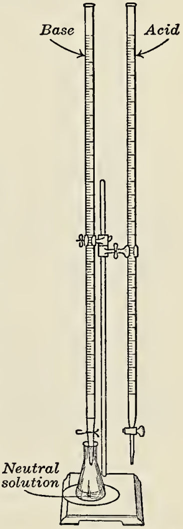

Fig. 41. Titration of a base with an acid.

e. Read the level of the liquid in each burette to one-tenth of a cubic centimeter (0.1 cc.) and record in the table. To find the volume of base used, subtract the first burette reading (before neutralization) from the burette reading after neutralization and *record in your notebook that result in the column of the table marked Difference.*

TABLE

Titration	Base Burette		Acid Burette	
	Readings	Difference	Readings	Difference
No. 1				
2				
3				

In a similar way find the volume of acid used to neutralize the base. Since the volumes of base and acid used are *inversely* proportional to the concentrations of the base and acid, we can easily compute the concentration of the acid (assuming the base to be 0.2 N):

$$\frac{x\text{ N}}{0.2\text{ N}} = \frac{\text{Volume base used}}{\text{Volume acid used}}$$

Wash out the flask and make another determination; repeat the process until you get three consistent results. *Take the average, or mean, of these three results as the concentration of the acid,* expressed as a decimal fraction of a normal solution.

EXPERIMENT 21

Conductivity of Substances in Solution

(*Class Experiment*)

Apparatus. — Conductivity apparatus (wide-mouth bottle fitted with 2-hole rubber stopper and carbon rods inserted). Lamp socket and 25-watt bulb. Electric current from lighting system or battery.

Materials. — Solutions of hydrochloric acid, sodium chloride, sodium hydroxide, alcohol, sugar, acetic acid, and ammonium hydroxide.

Introduction. If the laboratory is provided with electric lights (110 volts) suspended over the tables, it is easy to remove a lamp bulb and insert in its place a plug with extension cord. Connect the electrodes (platinum or carbon) of your conductiv ity apparatus (Fig. 42) in series with the socket of the lighting circuit. Screw a lamp (25–40 watts) into the socket of your apparatus and test your electric circuit by short-circuiting the two electrodes with a knife-blade. The lamp should glow brightly; in fact, the brilliancy of its light will measure roughly the electrical conductivity of the short-circuiting substance. Before testing the conductivity of any given solution, rinse the electrodes with distilled water until no sign of a glow is seen in the lamp filament when they are held for 10 seconds or more immersed in distilled water.

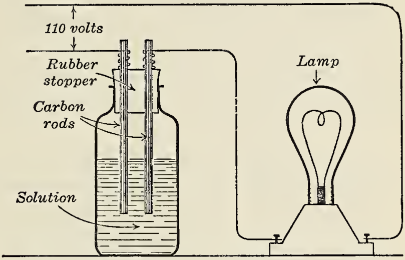

Fig. 42. Testing the conductivity of solutions.

Note. In cases where the electric-lighting circuit cannot be used, 4 dry cells or a 6-volt storage battery with a 6-volt miniature lamp as an indicator can be employed.

Electrolytes and nonelectrolytes. In setting up your conductivity apparatus and in changing the solution, *loosen the lamp in the socket of your apparatus and do not screw it down to make contact until all the connections have been made.* Test the conductivity of the following solutions: (1) dilute hydrochloric acid (1:4); (2) sodium chloride (10%); (3) sodium hydroxide (10%); (4) alcohol (10%); (5) sugar (10%); (6) distilled water.

Classify the substances used above according to their electrical conductivities.

Relative conductivity of acids. Prepare solutions of hydrochloric acid and acetic acid which have the same molecular concentration; for example, one-fifth normal (0.2 N). *Compare the conductivity of these acid solutions as indicated by the brightness of the lamp. Why is it necessary to have the electrodes im-*

mersed to the same depth in both cases? Which acid is more completely ionized?

Relative conductivity of bases. Prepare solutions of sodium hydroxide and ammonium hydroxide which have the same molecular concentration; for example, one-fifth normal (0.2 N). *Compare the conductivity of these basic solutions as indicated by the brightness of the lamp.* Arrange the electrodes so that they are immersed to the same depth in each case. *Which base is more completely ionized?*

Ions of acids and bases. *What ion is common to all acids? What is the other ion of hydrochloric acid? Of acetic acid?*

What ion is common to all bases? What is the other ion of sodium hydroxide? Of ammonium hydroxide?

What is the significance of the terms "strong" and "weak" as applied to acids and bases?

EXPERIMENT 22

Preparation of Soluble Salts

Apparatus. — Test tubes and rack. Tripod or ring stand and asbestos gauze. Bunsen burner. Funnel. Stirring rod.

Materials. — Dilute hydrochloric, nitric, and sulfuric acids. Zinc. Magnesium oxide. Sodium carbonate. Dilute solutions of sodium hydroxide and potassium hydroxide. Litmus papers. Filter paper.

1. Metal and acid. Many of the soluble salts (see Table of Solubilities in Appendix) of the common metals, such as zinc (Zn), iron (Fe), magnesium (Mg), and copper (Cu), can be prepared by replacing the hydrogen in the common acids. Such salts as zinc chloride ($ZnCl_2$), magnesium sulfate ($MgSO_4$), copper nitrate ($Cu(NO_3)_2$), and iron sulfate ($FeSO_4$) can be thus prepared.

To 5 cc. of dilute hydrochloric acid add zinc until there is no further action. Filter, evaporate a few drops of the filtrate almost to dryness on a watchglass, and cool. *What is the residue? Write the equation for the reaction.*

2. Metallic oxide and acid. Dissolve about 5 g. of magnesium oxide (MgO) in dilute sulfuric acid and filter if necessary; then evaporate a little of the clear solution to a small volume and cool. *What is the residue? Write the equation for the reaction.*

3. Carbonate and acid. Heat 10 cc. of a solution of sodium carbonate (Na_2CO_3) to the boiling point and add dilute hydrochloric acid as long as it causes effervescence. Evaporate a little of the resulting solution to dryness. *What is the residue? Write the equation for the reaction.*

Try the action of hydrochloric acid on solid sodium carbonate. *Compare the action of hydrochloric acid on a carbonate with the action of sulfuric acid on a chloride.*

4. Neutralization of a base and an acid. Sodium chloride (NaCl) has already (Exp. 19) been prepared by neutralizing sodium hydroxide (NaOH) with hydrochloric acid. In a similar way potassium chloride (KCl), sodium sulfate (Na_2SO_4), potassium sulfate (K_2SO_4), sodium nitrate ($NaNO_3$), and potassium nitrate (KNO_3) can be prepared.

Make one of these salts by neutralizing a suitable base and acid. Evaporate the salt solution nearly to dryness and examine the residue. *Write the equation for the reaction.*

5. ***Summarize** the four methods of making soluble salts.*

EXPERIMENT 23

Preparation of Insoluble Salts — Types of Reactions

Apparatus. — Test tubes and rack. Small beakers. Asbestos gauze. Bunsen burner. Tripod.

Materials. — Solutions of lead nitrate and lead acetate. Lead oxide (litharge) and lead carbonate. Sodium chloride solution. Dilute hydrochloric acid.

We may illustrate the various ways of preparing an insoluble salt by preparing lead chloride ($PbCl_2$) as follows:

1. Salt and acid. Heat in a test tube 20 cc. of lead nitrate ($Pb(NO_3)_2$) solution to boiling and add 5 cc. of dilute hydrochloric acid. Set aside to cool. *What is the precipitate? Write the equation for the reaction.*

2. Salt and salt. Heat 20 cc. of lead acetate ($Pb(C_2H_3O_2)_2$) solution and add 5 cc. of sodium chloride solution. Set aside to cool. *What is the precipitate? Write the equation.*

3. Oxide and acid. Dissolve in a beaker 1 g. of lead oxide (PbO) in boiling dilute hydrochloric acid and add about 50 cc. of boiling water. Decant the clear solution and set it aside. *What is the precipitate? Write the equation for the reaction.*

4. Salt and acid. Dissolve 1 g. of lead carbonate ($PbCO_3$) in boiling hydrochloric acid and proceed as in part (**3**).

Types of reactions. Chemical changes may be classified as (1) direct combination, (2) simple decomposition, (3) simple replacement, and (4) double replacement. The fourth class (double replacement) has been subdivided into three types according to the products: (*a*) one product **insoluble,** (*b*) one product **volatile,** and (*c*) one product **slightly ionized.** *Classify all the chemical reactions in this experiment and in the preceding one.*

EXPERIMENT 24

Forms of Sulfur

Apparatus. — Test tubes and rack. Crystallizing dish. Bunsen burner. Magnifying glass. Dish of water. Clamp. Funnel. Beaker.

Materials. — Roll sulfur. Carbon bisulfide. Filter paper (10 cm. in diameter). Dime or other piece of silver.

Introduction. Sulfur is a very interesting element, not only because it can easily be obtained in the three states — solid, liquid, and gaseous — but also because it exists in two solid, two liquid, and three vapor forms. The two solid forms differ in certain physical properties. One of them is **rhombic** in crystalline form and is stable at ordinary temperatures. At 96° C. it changes into another solid form, which is monoclinic, or **prismatic,** and is the stable form from that temperature up to its melting point, 119.25° C. If either solid form is heated slowly and carefully, it will give one of the liquid forms of sulfur; but if it is heated much hotter, it changes into an entirely different liquid form of sulfur, which boils at 445° C. The vapor of sulfur can exist in three different molecular forms (S_8, S_6, and S_2) according to the temperature. In this experiment you will study solid and liquid sulfur in its various allotropic forms.

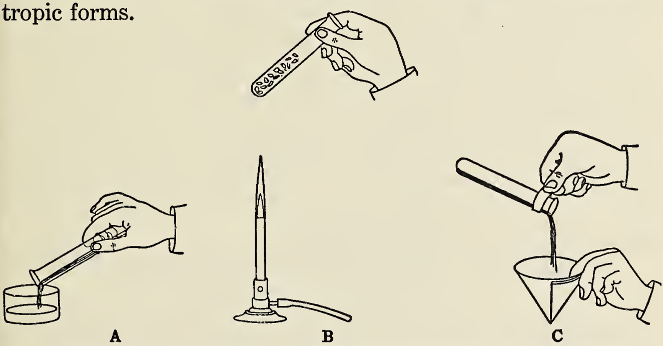

Fig. 43. A, Pouring a solution of sulfur into a crystallizing dish. B, Melting sulfur in a test tube. C, Pouring melted sulfur into a filter paper.

Directions. a. *Crystals obtained from solution.* Put about 2 or 3 g. of powdered roll sulfur in a test tube, add about a quarter of a test-tubeful of carbon bisulfide, and shake the tube.

Caution. Carbon bisulfide is a volatile liquid which easily takes fire. No flame should be near it.

Pour off the clear solution (Fig. 43 A) into a crystallizing dish. Set it aside to evaporate the liquid at some distance from a flame.

(While waiting for the crystals to form, proceed with part (b).)

When crystals have formed, examine them with a magnifying glass and note their shape; put them in a dry test tube and label them **rhombic sulfur.** *Draw several crystals in your notebook.*

b. *Crystals obtained by slowly cooling molten sulfur.* Place a folded filter paper in a funnel and have a dish of water at hand. Fill a clean, dry test tube half full of powdered sulfur. Melt the sulfur very slowly by holding the test tube in an inclined position well above the flame (Fig. 43 B) and by turning the tube in order not to overheat the sulfur at any point. The liquid sulfur should be pale yellow; if it becomes dark in color, it shows that it has been overheated.

Pour the liquid sulfur into a folded filter paper (Fig. 43 C) and watch the crystals form. As soon as they extend from the sides to the center of the cone, break the surface crust and pour the sulfur which is still liquid into the water in the dish. At once open up the filter paper and examine the crystals with a lens. *Make a drawing of a few of them and record their color and transparency.* This form of sulfur is called **prismatic sulfur.**

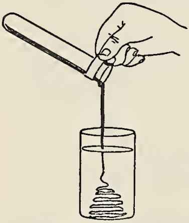

Fig. 44. Pouring boiling sulfur into cold water.

Keep some of these crystals and observe any change in their appearance.

c. *Plastic sulfur, obtained by heating sulfur to its boiling point and then suddenly cooling it.* Fill a beaker with water and place it on the table beside you. Half fill a test tube (preferably the one used in (b)) with small pieces of roll sulfur, hold the test tube in some kind of clamp (a folded strip of paper will serve), and very gently heat the sulfur until it melts. When it is completely melted, heat it more strongly and observe the changes in color and fluidity.

When the sulfur is boiling vigorously, pour it into the beaker of water (Fig. 44).

Caution. Do not become alarmed if the sulfur vapor begins to burn at the mouth of the test tube. Do not, however, spill burning sulfur on the table.

Observe what happens to the sulfur which remains in the test tube as it slowly cools.

Take the solid from the beaker and examine it. *Note its color. Is it hard or soft? Is it elastic or brittle?* This form of sulfur is called **plastic sulfur** or amorphous sulfur.

Find out whether plastic sulfur is soluble or insoluble in carbon bisulfide.

d. Test for free sulfur. Sulfur combines very readily with silver to form a black silver sulfide. Heat a minute particle of sulfur on a silver coin; a black stain is produced.

EXPERIMENT 25

Hydrogen Sulfide

Apparatus. — Ring stand and clamp. Large test tube (8″ × 1″) and 1-hole stopper to fit. Test tubes and rack. Bunsen burner. Glass tubing. Wide-mouth bottle. Glass nozzle.

Materials. — Iron sulfide. Dilute hydrochloric acid. Solutions of lead nitrate, copper sulfate, and cadmium nitrate. Litmus papers.

Introduction. The odor characteristic of rotten eggs is due to hydrogen sulfide, a gas much used in the chemical laboratory. It is indispensable to the chemist as a laboratory reagent in the processes of analysis. The common metals unite with sulfur to form sulfides, which vary in their solubility to such an extent that one may easily divide the metals into groups for final identification. These sulfides are often colored and their colors give us another means of identifying metals.

Besides being unpleasant, the gas is poisonous and produces headache when inhaled in considerable quantities. Therefore a water solution is often used in the laboratory.

Preparation. When iron filings and powdered sulfur are mixed together and heated, the product is iron sulfide (ferrous sulfide). Carefully slide two or three small pieces of iron sulfide (FeS) into a large test tube and pour in enough dilute hydrochloric acid to cover the iron sulfide.

Complete in your notebook the equation:

$$FeS + 2\,HCl \longrightarrow \text{------} + \text{------} \uparrow$$

Note the odor of the gas. Do not inhale much of it as it is somewhat poisonous.

Tests. The odor is a sufficient test when considerable quantities of the gas are present; but a more sensitive test is to dip a strip of filter paper into a solution of lead nitrate $Pb(NO_3)_2$ and to hold it in the gas to be tested.

Complete in your notebook the equation for testing for the gas with lead nitrate:

$$H_2S + Pb(NO_3)_2 \longrightarrow \text{------} \downarrow + \text{------}$$

Properties. **a.** To make a simple **hydrogen sulfide generator,** fit the large test tube with a 1-hole stopper and delivery tube as shown in figure 45. Collect the gas in a dry test tube by the displacement of air, remove the delivery tube, and light the gas in this test tube. As soon as the gas in the test tube burns quietly, attach a glass nozzle* or jet to the delivery tube and set fire to the gas.

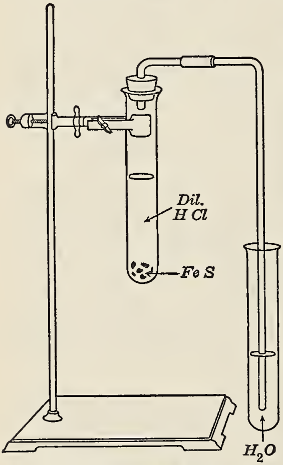

Fig. 45. Preparing a solution of hydrogen sulfide.

b. Cautiously smell the gas around the flame. Hold a cold, dry bottle over the flame and note whether any product of the combustion is condensed therein. *What are the two products of the complete combustion of hydrogen sulfide? Write the equation for this reaction in your notebook.*

c. Extinguish the flame and insert the delivery tube to the bottom of a test tube which is three-fourths full of water. Let the gas bubble through the water 3 or 4 minutes. (If the action in the generator slackens, gently warm the large test tube.)

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* A glass nozzle or jet is made by heating a straight piece of glass tubing in the Bunsen flame and, when soft, pulling it out so that the bore is reduced. The tube is then cut off at the constricted portion.

What effect has this solution on blue litmus paper? This solution is called **hydrosulfuric acid.**

Uses. A solution of hydrogen sulfide is a very useful reagent in chemical analysis to determine what metals are present in a material of unknown composition. Some of the metallic sulfides are insoluble and have distinctive colors.

Add a portion of the hydrosulfuric acid just prepared to a solution of copper sulfate. *What are the products formed? Complete the equation in your notebook:*

$$H_2S + CuSO_4 \longrightarrow \text{------} \downarrow + \text{------}$$

Add another portion of the hydrosulfuric acid to a solution of cadmium nitrate. *What are the products formed? Complete the equation in your notebook:*

$$H_2S + Cd(NO_3)_2 \longrightarrow \text{------} \downarrow + \text{------}$$

e. *Summarize the observed properties of hydrogen sulfide.*

EXPERIMENT 26

Sulfur Dioxide and Sulfurous Acid

(*Class Experiment*)

Apparatus. — Platinum wire. Bunsen burner. Bulb tube. Ring stand with ring and clamp. Asbestos gauze. Erlenmeyer (conical) flask (250 cc.) with 2-hole stopper. Funnel. Pinchcock (screw). Rubber connection tube. 3 wide-mouth bottles and glass squares. Test tubes. Evaporating dish. Glass tubing.

Materials. — Sulfur. Iron pyrite. Sodium bisulfite. Dilute hydrochloric acid. Pink flower. Thin slice of apple. Litmus papers. Zinc. Lead nitrate test paper.

Introduction. Everyone is more or less familiar with the choking gas known as "the smell of burning sulfur." This gas is sulfur dioxide (SO_2) and is formed when sulfur is burned or when a metallic sulfide is heated strongly. A more convenient method of preparing it in the laboratory is by slowly dropping hydrochloric acid upon sodium hydrogen sulfite ($NaHSO_3$). This experiment should be done under a hood.

Preparation. **1.** Touch a bit of sulfur with a warm platinum wire and bring the wire with adhering sulfur again into the flame. Withdraw it and *observe the color of the flame of burning sulfur; also note the odor of the gas produced. Write in your notebook the equation for the reaction.*

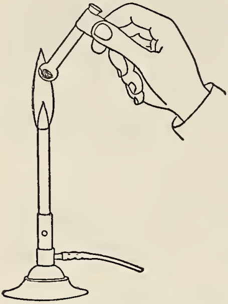

Fig. 46. Heating iron pyrite in a bulb tube.

Heat (or roast) in a little bulb tube (Fig. 46) a few particles of iron pyrite (FeS_2). *What is the solid which condenses on the cool part of the tube?*

2. A convenient method of preparing sulfur dioxide in the laboratory is to drop hydrochloric acid slowly on sodium hydrogen sulfite ($NaHSO_3$). This reaction may be expressed by the following equation:

$$NaHSO_3 + HCl \longrightarrow NaCl + H_2O + SO_2 \uparrow$$

Arrange the apparatus as shown in figure 47. Place in the flask about 10 g. of sodium bisulfite (sodium hydrogen sulfite) and replace the stopper carrying the funnel tube and delivery tube. Attach a straight glass tube to the delivery tube in order to make it reach down to the bottom of a wide-mouth bottle.

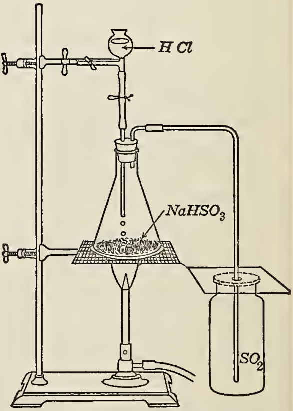

Fig. 47. Dropping hydrochloric acid on sodium bisulfite to generate sulfur dioxide.

Fill the funnel with dilute hydrochloric acid and then adjust the pinchcock to admit the acid into the flask drop by drop. To increase the speed of the reaction, warm the flask *very gently* from time to time as needed.

As soon as the air in the flask

has been swept out, collect two bottles of the gas by upward displacement of air. Cover each bottle with a glass plate. Boil half a test-tubeful of water in order to drive off the dissolved air and then cool it under running water. Insert the delivery tube to the bottom of the boiled water and let the gas bubble through for 5 minutes. Cork this test tube just as soon as the delivery tube is taken out in order to keep out the air.

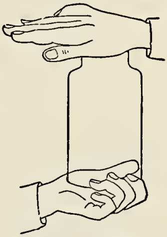

Fig. 48. Shaking a bottle containing sulfur dioxide and water.

Properties. **a.** Uncover one of the bottles of gas just enough to pour in a little water; then close it *tightly* with the palm of the hand and shake the contents (Fig. 48). Is the hand held against the mouth of the bottle by atmospheric pressure (suction) or not? *Is the gas soluble?*

b. In the second bottle of gas place a moist pink flower (carnation) and a thin slice of apple. Leave another slice of apple exposed to the air outside. Compare the pieces. *What is the effect of sulfur dioxide on vegetable coloring matter?*

c. Sulfurous acid. Test the aqueous solution made in (a) with litmus papers. *Write in your notebook the equation for the formation of sulfurous acid* (H_2SO_3). *Is sulfur dioxide an acidic or a basic oxide?*

Boil 5 cc. of this solution slowly in an evaporating dish and note the odor from time to time. *Write in your notebook the equation which represents this change and compare it with the preceding equation.*

To detect sulfurous acid, we may reduce it to hydrogen sulfide by using a suitable reducing agent, such as nascent hydrogen. Put a few pieces of zinc in a test tube and cover the metal with dilute hydrochloric acid. As soon as the action becomes vigorous, test the gas with a strip of moist lead nitrate paper (the paper should remain white). Now pour in 2 or 3 cc. of sulfurous acid and test again with lead nitrate paper.

d. *Summarize the properties of sulfur dioxide: color, odor, density as compared with air, and solubility.*

Also *summarize the properties of sulfurous acid: action on vegetable coloring matter; stability; oxidizing or reducing agent.*

EXPERIMENT 27

Sulfuric Acid

Apparatus. — Test tubes and rack. Bunsen burner.

Materials. — Concentrated sulfuric acid. Zinc. Dilute sulfuric acid. Sodium chloride. Wood splint. Dilute solutions of sodium sulfate, zinc sulfate, and copper sulfate. Barium chloride solution. Dilute hydrochloric acid.

Introduction. Sulfuric acid is the most important substance used in chemical industries, with the exception of water. The acid itself or some substance made by means of it is used in almost every industry. The manufacture of fertilizers, of explosives, and of other acids and the refining of petroleum oil are important examples of processes that use immense quantities of sulfuric acid. Ordinary commercial sulfuric acid is known as "oil of vitriol." We shall see in this experiment that its properties are quite different in concentrated form and in dilute solution.

1. Heat of dilution. Fill a test tube about one-third full of water and then pour into it very gradually an equal volume of concentrated sulfuric acid. Touch the outside of the test tube and *record any change in temperature.*

Caution. Remember that the concentrated sulfuric acid is to be poured slowly *into* the water. The reverse order produces dangerously explosive spattering. *Why?*

2. Action on zinc. Place a strip of zinc in a test tube and cover it with concentrated sulfuric acid. If there is no action, warm the acid cautiously.

CAUTION. Hot concentrated sulfuric acid causes very severe burns when it touches the skin. Do not pour hot concentrated sulfuric acid into water. Let it cool first.

Place another strip of zinc in a test tube and cover it with dilute sulfuric acid. *Name the products formed in this reaction and write in your notebook the equation.*

Compare the action of concentrated and of dilute sulfuric acid on zinc.

3. Action on common salt. Try the effect of both dilute and concentrated sulfuric acid on common salt (sodium chloride). *Identify the gaseous products.*

4. Action on wood and paper. Insert a wood splint into a little concentrated sulfuric acid in a test tube and let it stand for a few minutes.

Dip a glass rod into dilute sulfuric acid and write with it on paper. Warm the paper gently over a flame. *What property of sulfuric acid do these experiments with wood and paper illustrate?*

5. Test for a sulfate. Prepare in separate test tubes dilute solutions of *sulfuric acid, sodium sulfate, zinc sulfate,* and *copper sulfate.* Add to each solution a few drops of barium chloride solution ($BaCl_2$) and then dilute hydrochloric acid. *Complete in your notebook the equation:*

$$H_2SO_4 + BaCl_2 \longrightarrow \text{------} \downarrow + \text{------}$$

What insoluble precipitate is formed in each case?

To a solution of sodium phosphate add a few drops of barium chloride and dilute hydrochloric acid. *Why is it necessary to add hydrochloric acid in testing for sulfates?*

How would you distinguish between a sulfide, a sulfite, and a sulfate?

EXPERIMENT 28

Destructive Distillation of Coal

Apparatus. — Ring stand with clamp. Ignition test tube ("Pyrex") with 1-hole stopper. Large test tube with 2-hole stopper. Bunsen burner with wing top. Glass tubing.

Materials. — Small pieces of bituminous (soft) coal.* Litmus papers. Lead nitrate test papers.

Introduction. The coal we burn today was once living vegetation and long ago was buried in beds of sand. In consequence the vegetable matter has undergone a partial decomposition which has yielded amorphous carbon as the main product. Hard coal contains about 90 per cent carbon, nearly all of which is in the uncombined state; but soft coal contains considerably less uncombined carbon, and considerably more carbon combined with hydrogen, oxygen, nitrogen, and sulfur. Large quantities of soft coal are heated in iron retorts, or ovens, and the various products are collected and utilized. For example, ammonia gas is got from the **scrubbing towers**; a tarry matter, called "coal tar," is also saved and has proved to be an extremely valuable by-product. The final product is illuminating gas, which is now used extensively as a fuel. Coke is left in the retorts. Coke is not only a fuel but also a valuable aid in various metallurgical processes. In this experiment we shall carry out on a small scale the process of the destructive distillation of coal.

Distillation of coal. Fill an ignition tube about three-fourths full of small pieces of soft coal and clamp it horizontally, as shown in figure 49. Have the delivery tube extend nearly to the bottom of the condensing tube (large test tube). Heat the ignition tube gently at first and then strongly. During the heating, test the stream of gas in the delivery tube for hydrogen sulfide (H_2S) with a strip of paper moistened in lead nitrate solution, and also for ammonia with moistened red litmus paper. *Results?*

---

* The instructor should test the coal for ammonia before giving it out because some coal does not give off much ammonia.

Products. **a.** Bring a flame to the end of the jet tube. *Does the gas burn?*

b. Observe the properties of the material in the condensing tube. *Name it.*

c. When no more gas is given off, stop heating and allow the ignition tube to become cool enough to handle. Shake its contents out on the base of the ring stand. *Compare this residue in color, structure, and weight with the original coal. Name it.*

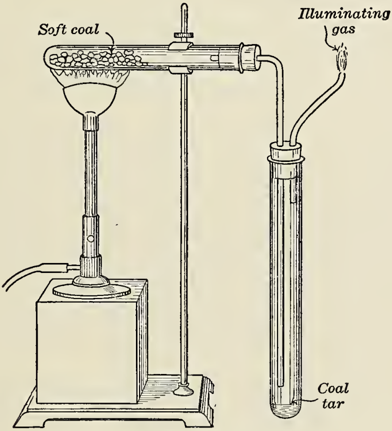

Fig. 49. Destructive distillation of coal.

d. *Why is this process called destructive distillation?*

e. *Name three important products of the destructive distillation of coal. Name a by-product.*

EXPERIMENT 29

Analysis of Coal — Gravimetric

(*Optional Experiment*)

Apparatus. — Ring stand with ring. Pipe-stem triangle. Porcelain crucible with cover. Bunsen burner. Horn-pan balance and weights. Mortar and pestle. Drying oven.

Materials. — Samples of coal (from home).

Introduction. When coal burns, the most evident constituents are (1) moisture, (2) volatile matter, (3) fixed carbon, and finally, (4) a residue of ash. The fixed carbon is volatilized only when heated in contact with air. These several constituents cannot be sharply separated, and hence such an analysis depends upon more or less arbitrarily standardized methods. This "proximate analysis" does, however, furnish an acceptable indicator of the type of coal. Coals are classified according to the relative proportion of fixed carbon to volatile matter. The ratio of fixed carbon to volatile matter is called the "fuel ratio." Thus the fuel ratio of anthracite coal is not less than 10; while the fuel ratio of bituminous coals varies usually from 3 to 6.

Direction. **a.** ***Moisture.*** Pulverize several pieces of coal to particles about the size of a pea and mix thoroughly. Take about a teaspoonful of this pulverized coal and grind it in a mortar to a very fine powder. Weigh to a centigram (0.01 g.) a clean, dry crucible and cover, and weigh out about 2 g. of the powdered coal. Record all these weights in tabular form:

Wt. of crucible and cover . . . . g.
Wt. of crucible, cover, and coal . . . . g.
Wt. of coal . . . . g.

Heat the crucible (uncovered) in an oven for an hour at 104°–107° C. Then cool and again weigh the crucible (including the cover). The loss of weight equals the weight of the moisture driven off. *Calculate the percentage of moisture in the coal.*

b. ***Volatile matter.*** Heat the covered crucible and contents from (a) on a pipe-stem triangle with the Bunsen flame until no more smoke comes off. Remember that a crucible should not be heated or cooled too suddenly, but when once hot it should be heated until the entire crucible is red-hot. Let the crucible cool with the cover on and then reweigh. The loss in weight during this heating is due to the volatile matter which has been expelled. *Compute the percentage of volatile matter in the coal.*

c. ***Fixed carbon*** (***coke***). Remove the cover from the crucible and incline it on the pipe-stem triangle (Fig. 50). Heat the crucible and contents from (b) to a full red heat until all the carbon has burned off. In the same way burn off any deposit on the cover. Cool and weigh the crucible (with cover) and its residue of ash. The loss of weight during this heating is the weight of the fixed carbon. *Calculate the percentage of fixed carbon in coal.*

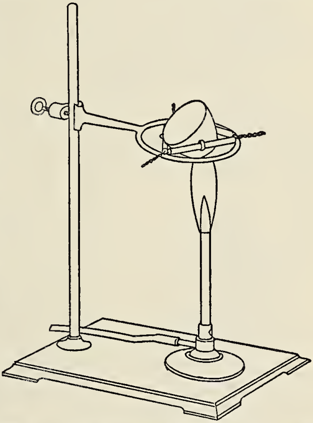

Fig. 50. Determining the ash in coal.

d. ***Ash.*** The residue left in the crucible from (c) is the nonvolatile mineral matter of the coal and is called ash. *Calculate the percentage of ash in coal.*

e. ***Compare*** the proximate analysis of your sample of coal with the analysis given in Engineering Handbooks (MARKS'S *Mechanical Engineers' Handbook*).

EXPERIMENT 30

Charcoal — Preparation and Properties

Apparatus. — Tripod. Bunsen burner. Sand crucible. Iron crucible and cover. Pipe-stem triangle. Ignition tube with 1-hole stopper. Ring stand and clamp. Test tubes. Mortar and pestle.

Materials. — Blocks of wood. Solution of molasses, brown sugar, cochineal, or indigo. Animal charcoal (boneblack). Copper sulfate solution. Hydrogen sulfide solution. Powdered copper oxide. Limewater.

Preparation. (*Class Experiment.*) When animal or vegetable matter is heated strongly in the absence of air, the residue is charcoal. We may easily prepare wood charcoal by placing on a thin layer of sand in a sand crucible or on a small iron pan a few chips or small blocks of wood. If we then cover them with sand to keep out the air (Fig. 51) and heat strongly until all smoking ceases, we find, when the crucible is cool, that the residue is charcoal.

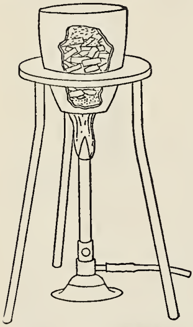

Fig. 51. Preparation of charcoal in a sand crucible.

Properties. (*Student Experiment.*) **a.** To show that carbon is a *decolorizer*, fill a test tube half full of water which has in it enough molasses, brown sugar, cochineal, or indigo to give a decided color. Add about 5 cc. of animal charcoal to the solution, shake thoroughly, and boil for 3–4 minutes. Filter the solution, and, if the filtrate is not clear and transparent, repeat the operation with fresh animal charcoal. The activity of the charcoal is much increased by previous heating in a covered iron crucible. If a solution of sugar or molasses was used, taste the filtrate to determine whether or not the sugar has been removed.

Repeat this experiment with a solution of copper sulfate. *Can the colors be removed from all liquids by filtering them through animal charcoal (boneblack)?*

b. To show that carbon is a *deodorizer*, fill a test tube about one-fourth full of powdered wood charcoal and add about 5 cc. of hydrogen sulfide (H_2S) solution. Cork the test tube and at short intervals for 15 minutes shake the contents vigorously. Compare the odor with that of the original solution and, if it is not perceptibly removed, add more charcoal and continue shaking the contents. *What use of this property of charcoal was made in the World War?*

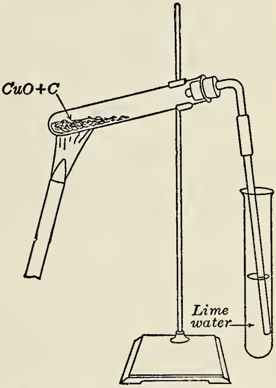

Fig. 52. Reducing copper oxide with carbon.

Uses. The most important chemical use of charcoal (or coke) is as a *reducing agent.* Mix intimately about 5 g. of copper oxide (CuO) with 1 g. of powdered wood charcoal in a mortar. By means of a folded strip of paper, slide the mixture into an ignition test tube (Pyrex) and fit it with a 1-hole stopper carrying a delivery tube. Arrange the apparatus as shown in figure 52 so that the gas evolved when the tube is heated will bubble through a little limewater in a test tube.

Heat the mixture in the ignition tube at first gently to avoid cracking the glass and then as strongly as possible, beginning at the part near the stopper and gradually moving the flame toward the closed end. The tube must be heated persistently for at least 10 minutes. *What is the first change that takes place in the limewater?*

Remove the delivery tube from the limewater and stop heating. When the tube is cold, pour its contents into a mortar and vigorously grind the residue under water. Let a stream of water wash away the lighter particles. *What substance is carried away by the water? What is left in the mortar? What element is taken from the copper oxide? Describe the process. Write in your notebook the equation of the reaction.*

Results. *Summarize the properties of charcoal (carbon) which you have observed in this experiment.*

EXPERIMENT 31

Carbon Dioxide — Preparation and Properties

Apparatus. — Wide-mouth bottle with 2-hole stopper. Thistle tube. 3 wide-mouth bottles and glass plates. Glass and rubber tubing.

Materials. — Marble chips. Dilute hydrochloric acid. Litmus paper (blue). Tapers. Limewater. Wood splint.

Introduction. Carbon dioxide is one of the gases which is always present in the atmosphere. It is also found in certain natural mineral-spring waters. We are all familiar with it as the gas which makes the foam in soda-water drinks. Since this gas is not combustible, it is a common ingredient of fire-extinguishing liquids. Wherever coal or fuels containing carbon are burned, one of the products is carbon dioxide. In fact, we ourselves are furnaces, as it were, breathing out carbon dioxide. This we shall show in the experiment.

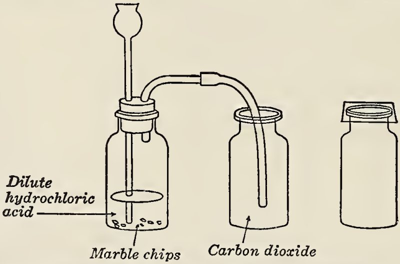

Fig. 53. Generating carbon dioxide and displacing the air.

Preparation. Use the same apparatus which you used to generate hydrogen, and arrange to collect 3 bottles of gas by displacement of air (Fig. 53). Slide enough marble chips into the bottle (don't drop them in) to cover the bottom. Add dilute hydrochloric acid through the thistle tube until a brisk evolution of gas is obtained. To test when a bottle is filled, bring a burning splint to its mouth; the gas will extinguish the flame. Cover each bottle with a glass plate. Allow the gas to bubble through a little water in a test tube. *Taste the liquid and test it with litmus paper. Complete in your notebook the equations:*

$$\underset{\text{marble}}{CaCO_3} + \text{--}\ HCl \longrightarrow \underset{\text{carbonic acid}}{H_2CO_3} + \text{----}$$

$$H_2CO_3 \longrightarrow \text{----} + \text{----}$$

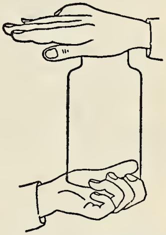

Fig. 54. Shaking carbon dioxide with water.

Properties. **a.** Light a taper and pour carbon dioxide from one of the bottles over it as you would pour water. *Does the gas support combustion? Is the gas heavier or lighter than air?*

b. Pour about 10 cc. of water into a bottle of the gas, cover at once tightly with the palm of the hand (Fig. 54), and shake it vigorously. *Is the hand sucked in, that is, held against the mouth of the bottle by atmospheric pressure? Is the gas soluble?*

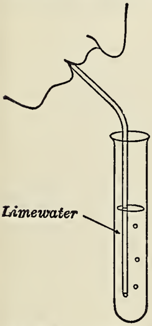

Fig. 55. Blowing through limewater.

c. Pour about half a test tube full of clear limewater into a bottle of carbon dioxide and shake the bottle. The milkiness is due to the calcium carbonate which is precipitated. This serves as a good **test for carbon dioxide.** *Complete in your notebook the equation:*

$$\underset{\text{limewater}}{Ca(OH)_2} + CO_2 \longrightarrow \underset{\text{calcium carbonate}}{CaCO_3} \downarrow + \text{----}$$

d. Burn a pine splint in a bottle of air. Add a little limewater and shake the bottle. *What gas is formed when wood burns?*

Blow through a glass tube into a test tube half full of limewater (Fig. 55). *What gas does the air exhaled from the lungs contain?*

Results. *Make a list of the properties of carbon dioxide which you have observed.*

EXPERIMENT 32

Carbon Monoxide — Preparation and Properties

Apparatus. — Ring stand with 1 ring and clamp. Wire gauze with asbestos center. Bunsen burner. Small flask with 2-hole stopper fitted with delivery tube and dropping funnel. Pinchcock (screw). Pneumatic trough. 3 wide-mouth bottles and glass squares.

Materials. — Concentrated sulfuric acid. Formic acid. Taper. Limewater.

Introduction. Another oxide of carbon is produced when any substance containing carbon burns in a *limited* supply of oxygen. This is called carbon monoxide. This gas, we shall see, is quite different from carbon dioxide in its properties. The most important fact to remember is that carbon monoxide is a poisonous gas. Exhaust gases of every gas engine contain some carbon monoxide. Very often we read of persons who have been poisoned while working on an automobile in a small closed garage. This gas also forms a large part of common illuminating gas, and for this reason one must never allow the gas to escape from the pipe unignited.

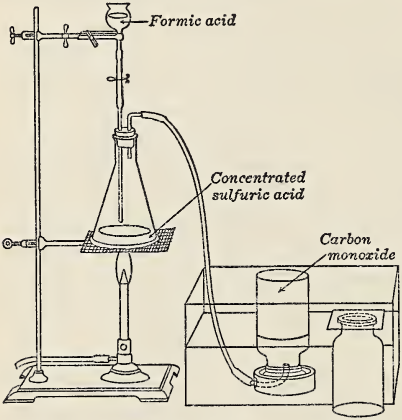

Fig. 56. Generating carbon monoxide by dropping formic acid on warm concentrated sulfuric acid.

Caution. **Carbon monoxide is very poisonous. Do not inhale it. Do not let it escape into the room. Get rid of any surplus gas by burning it.**

Preparation. Fit a small flask with a two-hole stopper carrying a delivery tube and a funnel connected by a short rubber tube and pinchcock with a straight tube, as shown in figure 56. Set up the apparatus on a ring stand with the asbestos wire netting under the flask at such a height as to heat the flask very gently with a small flame (Fig. 56). Arrange the delivery tube for collecting the gas over water. Remove the stopper from the flask and pour in 15 cc. of concentrated sulfuric acid. Replace the stopper and partially fill the funnel with formic acid (HCOOH). Never let this run out entirely during the experiment.

Allow a few drops of the formic acid to flow into the flask and warm very gently. Adjust the pinchcock to admit the formic acid one drop at a time and to get a steady flow of gas from the flask. Collect 3 bottles full of the gas and close the pinchcock to stop the generation of gas. *Complete in your notebook the equation:*

$$\underset{\text{formic acid}}{\mathrm{HCOOH}} \longrightarrow \text{------} \uparrow + \text{------}$$

Explain how the concentrated sulfuric acid helps along this simple decomposition.

Properties. a. Do not use the first bottle. *Why not?* Light a short taper and lift one bottle mouth downward. Thrust the burning taper up into it. *Does the gas burn? Does the taper burn in the gas?*

b. Take another bottle of the gas and slip the glass plate partly off in order to pour into the bottle 5 cc. of clear limewater; then quickly replace the plate and, holding it tightly against the mouth of the bottle, shake the contents of the bottle. *Note any change in the limewater.* Now light a taper, remove the glass plate, and quickly ignite the gas. Replace the cover at once and, when the flame dies out, shake the contents again. *What gas is formed when carbon monoxide burns?*

c. **Summarize** *the properties of carbon monoxide. How could you distinguish burning carbon monoxide from burning hydrogen?*

EXPERIMENT 33

Weight of a Liter of Oxygen

(Quantitative Experiment)

Apparatus. — Iron crucible. Ring stand and clamp. Pneumatic trough. 2-liter bottle (acid bottle). Test tube with 1-hole stopper. Horn-pan balance and weights. Bunsen burner. Glass plate. Graduated cylinder. Glass tubing.

Materials. — Manganese dioxide (powdered and thoroughly dried). Well dried (for several hours) potassium chlorate (c.p.). Glass wool or shredded asbestos.

Introduction. According to Avogadro's theory, equal volumes of gases under like conditions of temperature and pressure contain the same number of molecules. This gives a simple method of comparing the weights of molecules of gas by comparing the densities of the gases; that is, their weights per unit volume. Chemists have agreed to take the molecular weight of oxygen as exactly 32 and to use this as their standard of molecular weights. The density of oxygen has been determined with very great precision and is found to be about 1.429 grams per liter (0° C. and 760 mm.). This means that it takes $\frac{32}{1.429}$, or 22.4 liters of oxygen under standard conditions to weigh 32 grams. It is also found that if we divide the molecular weight of any gas by its density, we always get 22.4 liters. Therefore the most convenient way to determine the molecular weight of any gas is to find the weight in grams of 22.4 liters of the gas (at 0° C. and 760 mm.). Thus it is seen that the gram-molecular volume (22.4 l.) depends on the density of oxygen.

In this experiment we first determine the weight of a definite volume of oxygen and then compute the weight of one liter measured under standard conditions. To determine the weight of the oxygen, we weigh the generating apparatus both before and after a definite volume of oxygen has been produced. To measure the volume of the oxygen, we measure the volume of water which it displaces and then, knowing the temperature and barometric pressure, we compute according to the Gas Laws [**p. 592**] the volume under standard conditions (0° C. and 760 mm.).

Directions. a. It is important in this experiment to make sure that the loss of weight in the generating apparatus is due to the oxygen evolved and not in part to moisture driven off from the materials used. Therefore it is well to heat about 6 g. of pulverized manganese dioxide in an iron crucible (uncovered) for 5 minutes, with occasional stirring.

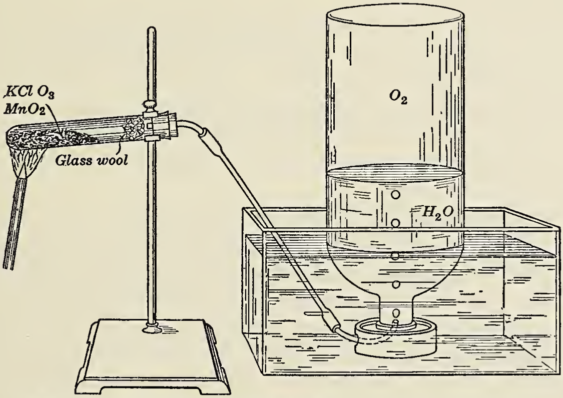

Fig. 57. Apparatus for finding the weight of a liter of oxygen.

In the meantime, fill an acid bottle (about 2 liters capacity) with water and invert it in the pneumatic trough. Insert a delivery tube in a one-hole rubber stopper which fits a dry test tube.

When the manganese dioxide has cooled, mix it on a sheet of paper with 7 g. of dry pulverized potassium chlorate. Slide the mixture into the test tube and place a plug of glass wool or well-ignited asbestos fiber in the mouth of the tube to catch small particles carried along by the gas. Weigh the test tube with its contents to the nearest centigram (0.01 g.). *Record the result in your notebook in a table.*

Replace the stopper in the test tube and test for air-tightness as follows. Place the end of the delivery tube in your mouth and suck out some of the air. If the tip of your tongue seems to adhere to the tube, the apparatus is tight. Arrange the apparatus as in figure 57.

b. Heat the contents of the test tube with a small flame, beginning at the top and gradually working downward. Collect all the gas evolved in the bottle. **Don't hurry! Regulate the heating so that you could count the bubbles of oxygen.**

When the bottle is nearly full, or when the oxygen has stopped coming over, remove the delivery tube from the water and allow the test tube to cool.

c. In the meantime, to measure the volume of the oxygen, first equalize the level of the water inside and outside the bottle. To do this, it may be necessary to lower the bottle and incline it. Then, while the bottle is in this position, close it with a stopper or glass plate and set it upright on the table.

To measure the volume of gas collected in the bottle, measure the amount of water needed to fill the bottle, pouring the water into the bottle from a graduated cylinder. *Record in your notebook the total volume of water used, the temperature of the water in the pneumatic trough, and the barometric pressure.*

When the test tube has cooled so that it feels barely warm, weigh the test tube and its contents as before and *record the weight in your notebook.*

Computation. Compute the loss of weight, which is the weight of oxygen evolved.

Correct the barometric pressure for aqueous tension [p. **597**] and then reduce the observed volume of oxygen from the observed temperature and pressure to 0° C. and 760 mm.

From this reduced volume and the weight, compute the weight of 1 liter of oxygen (density). *Do all your computations in your notebook.*

Table

Wt. of test tube + contents before heating	g.
Wt. of test tube + contents after heating	g.
Wt. of oxygen evolved	g.
Temperature	°C.
Volume of oxygen under conditions of exp.	cc.
Barometric pressure	mm.
Pressure due to water vapor	mm.
Corrected barometric pressure	mm.
Volume of oxygen under standard conditions	cc.
Density of oxygen (wt. of 1 liter)	g.

EXPERIMENT 34

Gram-Molecular Weight of Carbon Dioxide

(Quantitative Experiment)

Apparatus. — A 500-cc. flask with 1-hole stopper. Pinchcock. Rubber tube. Two wide-mouth bottles with 2-hole stoppers. Thistle tube. Horn-pan balance and weights. Ring stand. Glass tubing. Thermometer. Barometer.

Materials. — Marble chips. Dilute hydrochloric acid. Concentrated sulfuric acid.

Introduction. When the chemist uses as many grams of a substance as there are units in its molecular weight, he calls this weight the **gram-molecular weight.** Experiments show [p. **244**] that *the gram-molecular (molar) weight of any gas occupies under standard conditions 22.4 liters.* In the laboratory we weigh a given volume of gas (reduced to standard conditions) and then compute the weight in grams of 22.4 liters.

Directions. a. Arrange a large flask with a 1-hole rubber stopper which has a glass tube extending nearly to the bottom of the flask. Attach to the outer end of the glass tube a short rubber tube provided with a pinchcock. Weigh the flask fitted with stopper, tube, and pinchcock with great care to a centigram (0.01 g.). Then open the pinchcock and connect the rubber tube to a supply of dry carbon dioxide. This can easily be obtained by putting a few pieces of marble in a generating bottle and connecting it with a gas-washing bottle which contains concentrated sulfuric acid (Fig. 58).

b. Add dilute hydrochloric acid to the marble, loosen the rubber stopper in the flask so that a free current of gas may pass through, and pass a steady stream of carbon dioxide through the apparatus for 10 minutes. Then insert the stopper as before and disconnect from the generator. Open the pinchcock for an instant so that the pressure inside the flask may be the same as outside, and weigh the flask to centigrams.

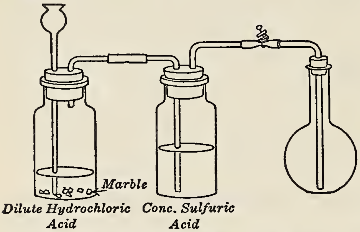

Fig. 58. Filling a flask with dry carbon dioxide.

To make sure that the flask is entirely filled with carbon dioxide, it is well to connect it again with the generator and to let the gas pass through for 5 minutes longer. As soon as constant weight has been attained (which indicates that the flask is entirely filled), read and record the temperature of the room and the barometric pressure.

c. The gain in weight of the flask when filled with carbon dioxide is, of course, only a small quantity because it is the difference between the weight of the carbon dioxide and the weight of the air in the flask. To find the weight of the air, we determine its volume by filling the flask with water from a graduated cylinder, reducing this volume to standard conditions and then computing the weight of that volume of air. (Assume that 1 liter of air at 0° C. and 760 mm. weighs 1.29 g.) To get the weight of the carbon dioxide in the flask, add the weight of the air in the flask to the gain in weight when filled with carbon dioxide. We now have determined the weight of a certain volume (reduced to standard conditions) of carbon dioxide and can readily compute the weight of 22.4 liters of the gas.

Arrange all your calculations in an orderly fashion in your notebook and record your data and results in tabular form:

Table

Weight of flask with air	g
Weight of flask with carbon dioxide, 1st trial	g.
Weight of flask with carbon dioxide, 2d trial	g.
Gain in weight of flask when filled with carbon dioxide . . .	g.
Temperature of room	° C.
Barometric pressure	mm.
Volume of the flask	cc.
Volume of flask at 0° C. and 760 mm.	cc.
Weight of air in flask (1 liter = 1.29 g.)	g.
Weight of carbon dioxide in flask	g.
Weight of 22.4 liters of carbon dioxide	g.

EXPERIMENT 35

Atomic Weight of Copper

(*Quantitative Experiment*)

Apparatus. — Ring stand and clamp. Ignition test tube ("Pyrex"). Horn-pan balance and weights. Bunsen burner. Glass tubing. Rubber tubing.

Materials. — Copper oxide (powder).

Introduction. We may determine the combining weight of copper by reducing a known weight of copper oxide to metallic copper. Assuming the combining weight of oxygen to be 8, we may compute from the actual weights of copper and oxygen the **combining weight of copper.** The atomic weight of copper is either equal to or some multiple of its combining weight. To determine this multiple we may make use of a fact discovered by Dulong and Petit: *the products of the atomic weights of the solid elements by their specific heats* are in almost all cases*

---

* Heat is measured in calories. A calorie is the heat required to raise the temperature of a gram of water 1° C. The number of calories required to raise the temperature of a gram of a substance 1° C. is called its **specific heat.**

numbers lying between 6 and 7, the average being 6.4. This is *not* a method of *determining* atomic weights but is an easy way to find the factor (*valence*) by which the combining weight must be multiplied in order to get the atomic weight.

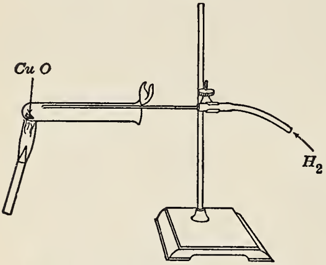

Fig. 59. Reducing copper oxide to copper with illuminating gas.

Directions. Clean and dry an ignition test tube and weigh it to a centigram (0.01 g.). Slide into the tube by means of a folded strip of paper 2 or 3 g. of black copper oxide (powder). Be sure that all of the oxide is at the bottom of the tube and carefully weigh the tube with the copper oxide. Clamp a piece of glass tubing in a horizontal position as in figure 59. Since illuminating gas contains a large percentage of hydrogen, it is convenient to use it as the reducing agent. We may burn the excess of gas as it issues from the mouth of the test tube and then turn the gas down so that the flame is only about 1 cm. high. Heat the test tube slowly until the oxide is red-hot. After 5-minutes' heating, turn off the gas and rotate the test tube, tapping it to make the oxide fall to the other side. Again insert the gas tube and continue heating until the black copper oxide is entirely reduced to red copper. Cool the ignition tube and weigh again.

Computation. The loss of weight is the weight of the oxygen removed from the oxide. Assuming the combining weight of oxygen to be 8, *calculate the combining weight of copper;* thus:

$$\frac{x}{8} = \frac{\text{Wt. of copper}}{\text{Wt. of oxygen}}$$

Assume the specific heat of copper to be 0.0936 and the constant product (at. wt. × sp. ht.) to be 6.4; *compute the approximate atomic weight of copper. What is the factor* (whole number) *by which the combining weight must be multiplied in order to get the atomic weight?* Multiply the combining weight of copper by this factor. *The product is the atomic weight.* Compare this result with the value given in the Table of Atomic Weights and *compute the percentage error.*

Show clearly in your notebook all your computations and record your data and results in tabular form:

Table

Wt. of test tube	g.
Wt. of test tube + copper oxide	g.
Wt. of test tube + copper	g.
Wt. of oxygen	g.
Wt. of copper	g.
Combining wt. of copper (Oxygen = 8)	
Factor by which the combining wt. must be multiplied in order to get the atomic wt.	
Atomic wt. of copper	
Atomic wt. of copper according to Table of Atomic Weights	63.6
Percentage error	%

EXPERIMENT 36

Composition and Formula of a Compound

(Quantitative Experiment)

Apparatus. — Porcelain crucible and cover. Pipe-stem triangle. Tripod or ring stand with 1 ring. Bunsen burner. Horn-pan balance and weights.

Materials. — Copper wire (# 30). Sulfur (powdered).

Introduction. When metallic copper is heated with sulfur, the product is a compound, copper sulfide. If we know the weight of the copper and of the copper sulfide produced, we may compute the weight of the sulfur which has united with the copper. Then we can compute what fraction (percentage) of the copper sulfide is copper and what fraction is sulfur. Finally, using the percentages of composition just determined and the atomic weights (taken from the table), we can derive the *simplest* formula of copper sulfide.

In this experiment more sulfur will be used than is required to unite with the copper to form copper sulfide. The *excess* sulfur is vaporized, escapes from the crucible, and is burned to sulfur dioxide, which escapes up the hood. The success of this experiment depends on care in weighing and patience in following out the directions in every detail.

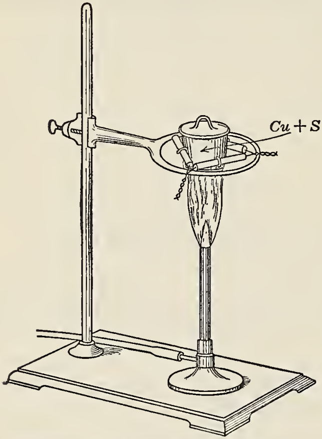

Fig. 60. Making copper sulfide.

Directions. a. Weigh a clean, dry crucible without the cover to the nearest centigram (0.01 g.). Wind about 2 meters of fine copper wire about a glass tube and place the coil in the crucible. The coil should be fastened together loosely. Weigh again carefully. Set the crucible on a pipe-stem triangle supported in such a position that it can be heated. Put about 1 gram of flowers of sulfur in the crucible and stir the contents. Now set the cover on the crucible and heat gently at first with a Bunsen flame (Fig. 60). Gradually increase the heat to the full capacity of the burner. Continue the heating in this way until the blue flames cease; then heat strongly for 2 minutes.

Allow the crucible to cool to room temperature. When it is cold (not before), remove the cover and reweigh the crucible and contents to the nearest centigram.

Questions:

1. *Why is it unnecessary to weigh precisely the sulfur?*
2. *Why should the crucible not be weighed while it is hot?*

Computation. Copy the following table and record your data and calculations as follows:

Wt. of crucible and copper wire . . . . g.
Wt. of crucible empty . . . . g.
Wt. of copper taken . . . . g.
Wt. of crucible and copper sulfide . . . . g.
Wt. of crucible empty . . . . g.
Wt. of copper sulfide . . . . g.
Wt. of sulfur used . . . . g.

$$\text{Percentage of sulfur} = \frac{\text{wt. of sulfur}}{\text{wt. of copper sulfide}} \times 100 = \ldots\ldots \quad \%$$

$$\text{Percentage of copper} = \frac{\text{wt. of copper}}{\text{wt. of copper sulfide}} \times 100 = \ldots\ldots \quad \%$$

b. *Find the simplest formula for copper sulfide.* To do this, divide the percentage of sulfur by its atomic weight, and also the percentage of copper by its atomic weight. These quotients will show the relative number of each kind of atom.

Percentage		At. Wt.		At. Ratio
Sulfur	÷	32	=	
Copper	÷	63.6	=	

What is the simplest formula for this sulfide of copper?

EXPERIMENT 37

Atmospheric Nitrogen

(Optional Experiment)

Apparatus. — A 2-liter bottle with 2-hole stopper. Hard-glass tubing (20 cm.) with two 1-hole stoppers. Bunsen burner. 3 wide-mouth bottles. Pneumatic trough. Ring stand and clamp. Tubing.

Materials. — Copper gauze or turnings.

Introduction. The atmosphere is composed of about four-fifths nitrogen, about one-fifth oxygen, and slightly less than one per cent argon together with varying amounts of carbon dioxide and water vapor and traces of rare gases. Since the air is a mixture composed of so many gases, it will be worth while to study in more detail one of them, atmospheric nitrogen.

Preparation of atmospheric nitrogen. Since the oxygen of the air combines readily with hot copper and the nitrogen does not, it is easy to obtain nitrogen from air. Set up the apparatus as in figure 61. The hard-glass tube contains copper gauze or clean copper turnings. The current of air is obtained by letting water from the tap fill the bottle.

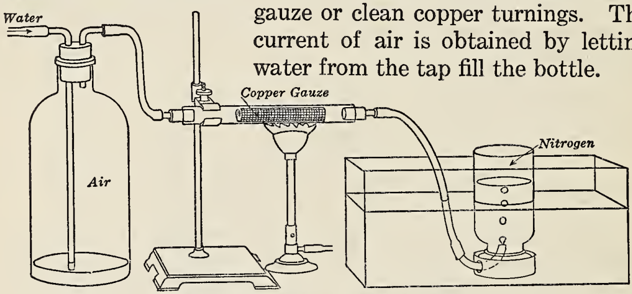

Fig. 61. Passing air over hot copper.

Heat the copper at first gently by moving the flame along the tube and then strongly. Do not burn the stopper. Carefully turn on the water in order to drive the air from the bottle very slowly over the hot copper. Collect three bottles of gas.

Properties. **a.** Apply a lighted taper to a bottle of the gas collected. *What happens?* Leave the bottle uncovered for 3 minutes and test again. *Does the taper now burn in the gas or not? Conclusions?*

b. Add a little clear limewater to another bottle and shake the contents. *Does the limewater turn milky?*

c. Find out whether burning sulfur will continue to burn in nitrogen.

d. *Make a list of the properties of nitrogen which you have observed.*

What is the black substance seen on the copper after cooling?

What component of the air was removed by the copper?

How can you distinguish nitrogen from carbon dioxide?

Why was the air passed over the copper ***slowly?***

What impurity does the nitrogen thus prepared contain? (p. **263.**)

How would you show the presence of moisture in the air? How remove it?

EXPERIMENT 38

Percentage of Oxygen in Air

(Quantitative Experiment)

Apparatus. — Pneumatic trough. Ring stand with clamp. 100-cc. graduated cylinder. Graduated test tube (25 cc.). Thermometer. Barometer.

Materials. — Yellow phosphorus. Copper wire (30 cm. # 18). Pyrogallol. Sodium hydroxide solution (1 to 4 by weight). Wood splint.

Introduction. A convenient method of determining what percentage of the air is oxygen consists in burning some substance in a definite volume of air and then measuring the volume of the remaining gas. Yellow phosphorus is a good substance for this purpose because it forms an oxide which is soluble in water.

Caution. Great care must be taken never to handle yellow phosphorus except under water.

Directions. a. To get a definite volume of air, invert a 100-cc. graduated cylinder so that its mouth dips under the surface of the water in a pneumatic trough. The cylinder may be tipped to let part of the enclosed air escape, and then it should be securely clamped in a vertical position so that the water level is the same inside and outside the cylinder. *The volume of the air thus confined* (about 100 cc.) *should be carefully observed and recorded.* Also *record the barometric pressure and the temperature of the water,* which should have stood long enough to be at the temperature of the room. Applying the Gas Laws, *compute the volume that this air would occupy under standard conditions (0° C. and 760 mm.).*

b. A piece of ordinary phosphorus should now be stuck on the end of a piece of copper wire * (under water), and the wire carrying the phosphorus should be pushed up into the air space in the cylinder and allowed to stand overnight. It is well to arrange the apparatus (Fig. 62) so that the phosphorus does not touch the sides of the tube and stands well up toward the top of the cylinder.

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* It will be safer to let the instructor fasten the wire around the phosphorus. Transfer the phosphorus to your desk in an evaporating dish, keeping it under water all the time. Return the phosphorus in the same way.

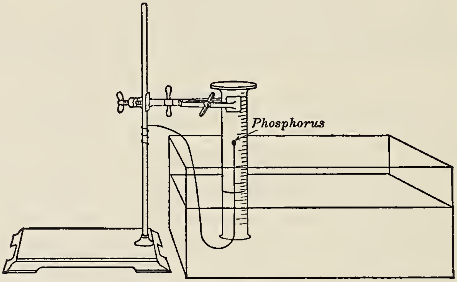

Fig. 62. Burning the oxygen out of air with phosphorus.

c. The next day remove the phosphorus from the cylinder and lower the cylinder until the level of the water inside is the same as that outside. Read and *record the volume of the gas remaining in the cylinder,* also *the temperature of the water and the barometric pressure.*

d. Compute the volume which the remaining gas would occupy if it were under standard conditions. Then calculate the difference between the original volume of air enclosed and the volume of gas that remains (both at standard conditions). This represents *the volume of oxygen* removed by the phosphorus.

Calculate the percentage of oxygen in air as follows:

$$\frac{x}{100} = \frac{\text{Volume of oxygen}}{\text{Volume of air}}$$

e. *What properties did you observe in the gases left after the oxygen had been absorbed? What were the gases?*

Make all calculations in your notebook and record your observations and the results of your calculations in tabular form:

Volume of air taken . . . . cc.
Temperature of the water, first day . . . . ° C.
Barometric pressure, first day . . . . mm.
Volume of air at 0° C. and 760 mm. . . . . cc.
Volume of gas remaining in cylinder . . . . cc.
Temperature of water, second day . . . . ° C.
Barometric pressure, second day . . . . mm.
Volume of gas remaining at 0° C. and 760 mm. . . . . cc.
Volume of oxygen absorbed by phosphorus . . . . cc.
Percentage of oxygen in air (by volume) . . . . %

Alternative method. Another and quicker method of absorbing the oxygen from the air is by means of an alkaline solution of pyrogallol. This is an organic chemical used as a photographic developer. It is impossible to write an equation representing the organic chemistry involved in this process because the products of oxidation are numerous and complicated. Although this method is often used in volumetric analysis, it requires considerable skill to get good results.

Directions. Fit a good, solid, rubber stopper to a 100-cc. graduated cylinder and measure with a centimeter-rule the distance between the top-graduation mark and the bottom of the stopper. By laying the rule alongside the graduations on the cylinder, determine the number of cubic centimeters between the top-graduation line and the bottom of the stopper.

Grind up about 1 gram of pyrogallol in a mortar and place the powder in the bottom of the graduated cylinder. Now measure out in a graduated test tube a volume of sodium hydroxide solution (1 to 4 by weight) equal to the volume of the cylinder between the graduations and the stopper. Pour this alkaline solution into the cylinder, which already has about 1 gram of pyrogallol in the bottom. Instantly replace the stopper firmly and shake the cylinder vigorously for two or three minutes. This alkaline solution of pyrogallol absorbs the oxygen from the air in the cylinder.

Invert the cylinder and thrust the top into a pneumatic trough nearly full of water at room temperature. Then remove the stopper. Note the result.

Allow the cylinder to stand in the trough of water for a few minutes until the cylinder and the gas have come to the temperature of the room. Equalize the water levels inside and outside the cylinder by raising or lowering the cylinder. *Note precisely the volume of gas left in the cylinder.* The difference between this volume and the 100 cc. of air is the volume of oxygen absorbed.

Again close the mouth of the cylinder with a stopper, remove it from the trough of water, and stand it upright on the table. Test the remaining gas in the cylinder with a burning splint. *Note the result.* The gas left is mainly nitrogen.

Record your data and results in your notebook in tabular form:

Data:
Volume of air taken . . . . 100.0 cc.
Volume of gas remaining in cylinder . . . . ______ cc.

Calculations: Volume of oxygen absorbed . . . . cc.
Percentage of oxygen in air . . . . %

EXPERIMENT 39

Ammonia — Preparation and Properties

Apparatus. — Large test tube (20 × 2.5 cm.) fitted with 1-hole stopper. Ring stand with ring and clamp. Bunsen burner. 3 wide-mouth bottles. Glass squares. Pneumatic trough. Glass tubing.

Materials. — Slaked lime. Ammonium chloride. Litmus papers. Tapers. Concentrated hydrochloric acid.

Introduction. When organic compounds containing nitrogen are heated with lime, they are decomposed and one of the products is ammonia (NH_3). This is the familiar gas which is characteristic of the household cleaning agent known as "ammonia water." But ammonia formed in this way is always mixed with large amounts of other substances, which are formed during the decomposition of the organic compounds. When coal is heated in a closed vessel and the gaseous products are passed through water, the ammonia gas dissolves. This ammonia solution is then mixed with sulfuric acid, and the ammonium sulfate is separated by crystallization. In the laboratory, ammonia can be readily prepared from any ammonium salt, such as ammonium sulfate ($(NH_4)_2SO_4$) or sal-ammoniac (NH_4Cl), by heating it with any of the alkalies. Since lime ($Ca(OH)_2$) is the cheapest alkali, it is the one commonly employed. As the gas, ammonia, is lighter than air, we may collect it in an inverted bottle by displacing the air.

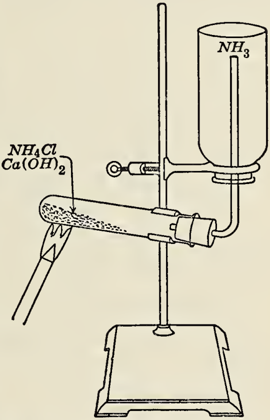

Fig. 63. Preparing ammonia and displacing the air.

Directions. a. ***Preparation.*** Mix on a piece of paper 2 parts of powdered slaked lime ($Ca(OH)_2$) and 1 part of ammonium chloride (NH_4Cl). Place this mixture in a large test tube which is provided with a delivery tube, as shown in figure 63. Do not fill the tube more than half full. Clamp and incline it slightly downward in order that any condensed moisture may not run back and crack the tube.

Warm the mixture very gently, beginning with the portion near the mouth of the tube and gradually extending the heat to other portions, but keep the flame moving continually. As soon as the smell of ammonia shows that the gas is escaping from the bottle, lift it and cover its mouth with a glass plate. Set it aside mouth downward. In this way collect 3 bottles of the gas.

b. ***Properties.*** Put one bottle mouth downward into a basin of water (pneumatic trough). Remove the glass plate and shake the bottle a little. *Note what happens.* Replace the glass plate and remove the bottle with its contents. Test the liquid with litmus papers. *What is the action of ammonia on water? Write in your notebook the equation.*

Pour into a warm, dry bottle two or three drops of concentrated hydrochloric acid. Cover this bottle with a glass plate and invert it upon a covered bottle of ammonia. Keep the bottles mouth to mouth while you remove both plates at the same time. The white solid which is formed is ammonium chloride (NH_4Cl). *Write in your notebook the equation for the reaction.*

Push a lighted taper up into an *inverted* bottle of the gas. *Does the gas burn? Does it support combustion of a taper?* Apply a lighted taper to the gas issuing from the exit-tube of the generator. *Observe whether the gas burns* (1) *while the lighted taper is present and* (2) *after it is removed.*

c. ***Results.*** Make a *list of the properties of ammonia* which you have observed in this experiment. Complete the equation for its preparation:

$$Ca(OH)_2 + 2\,NH_4Cl \longrightarrow \text{------} + \text{------} + \text{------}\uparrow$$

How could you prove that a given substance was an ammonium salt?

EXPERIMENT 40

Analysis of Household Ammonia

(Quantitative Experiment)

Apparatus. — 10-cc. pipette. Small beaker. Ring stand and clamp. Burette.

Materials. — Bottle of "household ammonia." Methyl-orange solution. Normal hydrochloric acid solution.

Introduction. The value of "household ammonia" depends upon the amount of base (NH_4OH) which it contains. It often happens that some brands contain one-tenth as much as others and yet are retailed at the same price. There is no way in which the average consumer can detect the difference, and often the grocer does not know the concentration of the brand he sells. However, it is quite easy for a student of chemistry to measure the concentration of a sample of household ammonia by titration with an acid of known concentration. Our first step is to review the experiment on the neutralization of a base and an acid by titration (Exp. 20).

Directions. Bring from home or buy of a grocer at least 100 cc. (4 oz.) of household ammonia. Take a 10-cc. pipette (Fig. 64) and rinse it out with the ammonia solution (throw away the rinsings). Measure out very carefully 10 cc. of the household ammonia with a pipette as follows: Insert the jet end of the pipette deeply in the liquid and place the other end in the mouth. By drawing (sucking) the air out of the pipette, the liquid is forced up into it. As soon as the liquid rises above the 10-cc. mark on the stem, remove the pipette from your mouth and quickly cover the end with your finger. By moving your finger slightly, the liquid is allowed to flow out until the surface is just on the mark.

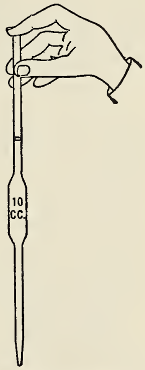

Fig. 64. A 10-cc. pipette.

Let this carefully measured volume of household ammonia flow out into a clean beaker and add about 20 cc. of distilled water. Add two or three drops of methyl-orange solution to the mixture. *This indicator turns red with acids and yellow with bases.*

Fill a burette with a normal solution of hydrochloric acid. Take the usual precautions about rinsing out the burette with a little of the solution to be used in it and see that the tip is filled with the solution. *Read and record the surface of the acid solution* (which must be on the graduated portion of the burette). Then place the beaker on a sheet of white paper directly under the burette and let the acid flow slowly into the ammonia solution, stirring constantly, until the change of color shows that enough acid has been added. In fact, too much acid has doubtless been used in this preliminary trial, but *note the volume of acid actually used.*

In the next trial the acid may be drawn off rapidly until the volume used has come within 2 cc. of that employed in the first trial. Now proceed very slowly until a single drop changes the color of the indicator. Make several trials until three consecutive trials agree within 0.5 cc. Find the average of these three consistent values for the volume of normal hydrochloric acid which neutralizes 10 cc. of the household ammonia.

Results. Compute the volume of normal acid required to neutralize 1 cc. of the ammonia solution. This volume, or **factor,** expresses the multiple of normality of the ammonia solution. If this volume is less than one, it expresses the fractional normality of the ammonia solution. A normal ammonia solution contains 17 g. of ammonia per liter, or is about 1.7 per cent ammonia. Therefore the product of the normality of the ammonia solution and 1.7 gives the percentage of ammonia in the household ammonia.

Arrange the data and results in tabular form and show each step in the computation.

EXPERIMENT 41

Nitric Acid

Apparatus. — Tubulated retort (250 cc.) with glass stopper. Tripod. Bunsen burner. Asbestos gauze. Ring stand and clamp. Large test tube. Battery jar. Funnel. Test tubes.

Materials. — Sodium nitrate. Concentrated sulfuric acid. Excelsior. White woolen yarn. Concentrated ammonium hydroxide. Copper turnings. Zinc (mossy). Ferrous sulfate solution (freshly prepared and saturated).

Introduction. Nitric acid boils at a much lower temperature than sulfuric acid and therefore can be prepared by the double decomposition between a nitrate and sulfuric acid. The mixture has to be heated hot enough to make the nitric acid pass off as fast as it is formed. But nitric acid very readily decomposes when it is heated, and so the acid is prepared at the lowest temperature which can be used to effect its production. Since nitric acid is very corrosive, the preparation must be carried on in apparatus composed entirely of glass.

Caution. **Nitric acid and sulfuric acid are both very corrosive. Do not drop any acid on your flesh or clothing.**

Preparation. Place about 20 grams of sodium nitrate on a piece of paper and fold the paper so that the solid may be slipped into the retort through the short tubular opening. Support the retort on the wire gauze with asbestos center, which is itself supported on a tripod. Clamp the neck of the retort in such a way (Fig. 65) that it extends into a large test tube or small flask partly immersed in cold water. Pour 15 cc. of concentrated sulfuric acid through a funnel upon the nitrate and then carefully remove the funnel without dropping acid from it. Replace the glass stopper in the short tube.

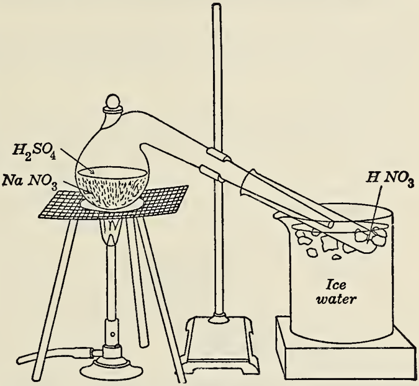

Fig. 65. Making nitric acid with a tubulated retort.

As soon as the sulfuric acid has moistened the entire mass of nitrate crystals, begin heating the retort gently. Distill at as low a temperature as possible until no more nitric acid condenses in the neck of the retort. The brown color observed in the retort is due to a little nitric dioxide (Exp. 42).

Allow the retort to cool in position. The residue, which is sodium hydrogen sulfate ($NaHSO_4$), may be removed with warm water. *Write in your notebook the equation for the preparation of nitric acid.*

Properties. 1. The nitric acid just prepared is 100% pure, and great care must be used in handling it. Pour 2 cc. of this acid into a test tube and insert a small, loose wad of excelsior so that it remains about an inch above the acid. Holding the test tube in a holder, boil the acid vigorously until the vapor reaches the excelsior. Now move the test tube so as to heat the excelsior itself for a few seconds. *How does this experiment illustrate the oxidizing action of nitric acid?*

2. Pour 2 cc. of the acid on a piece of white woolen yarn in a test tube, rinse with water, and then add ammonium hydroxide. This action of nitric acid followed by ammonium hydroxide serves as a test for those nitrogenous substances known as proteins.

3. Pour 2 cc. of the acid on a small piece of copper in a test tube. This action is characteristic of nitric acid. Repeat this experiment using zinc instead of copper. *Compare the action of nitric acid on metals with that of hydrochloric and sulfuric acids.*

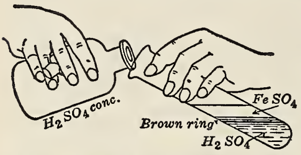

Fig. 66. Testing for nitrate ions with ferrous sulfate solution.

Test for the nitrate (NO_3^-) ion. This is a very delicate test, and the directions must be followed with care. Pour into a test tube about 3 cc. of ferrous sulfate ($FeSO_4$) solution. Add to it 2 drops of dilute nitric acid and shake the test tube. Holding the tube in an inclined position, pour 2 cc. of concentrated sulfuric acid slowly down the side of the tube (Fig. 66). The heavy acid will run down the tube and form a separate layer under the mixture of the other two solutions already in the tube.

Sometimes the test fails because the tube becomes hot. In this case let the tube stand quietly a few minutes. *Describe the coloration in the ring where the two layers meet.* Repeat this test using a very small amount of sodium nitrate dissolved in 2 cc. of water instead of the dilute nitric acid.

Summarize the properties of nitric acid.

EXPERIMENT 42

Nitric Oxide and Dioxide

Apparatus. — Wide-mouth bottle with 2-hole stopper. Thistle tube. Pneumatic trough. 3 wide-mouth bottles and glass squares. Deflagrating spoon. Large test tube with 2-hole stopper. Dropper. Glass tubing.

Materials. — Copper turnings. Concentrated nitric acid. Tapers. Asbestos paper. Red phosphorus. Sodium peroxide.

Introduction. Nitrogen forms five different oxides. However, only three of these, nitric oxide (NO), nitric dioxide (NO_2), and nitrous oxide (N_2O), are of sufficient importance to be studied at present. The action of nitric acid upon metals is more complicated than that of the other common acids, such as hydrochloric acid and sulfuric acid, which we have already studied. This is because it acts as a powerful oxidizing agent as well as an acid. When dilute nitric acid is allowed to react with metallic copper, the metal probably first displaces the hydrogen, as with other acids. But this hydrogen is immediately oxidized into water by the oxygen which is given off by the nitric acid. Therefore the final result is that we have copper nitrate ($Cu(NO_3)_2$) and water left in the flask, and nitric oxide is the gas produced. This gas readily changes into nitric dioxide on exposure to the air, as we shall see in the following experiment.

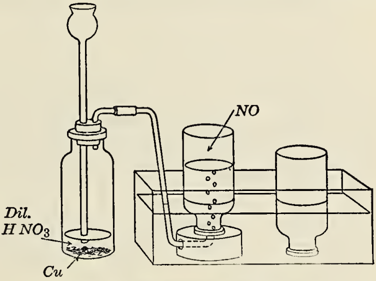

Fig. 67. Making nitric oxide.

Preparation. Place some pieces of copper (about 10 g.) in a generating bottle and fit it with a 2-hole stopper carrying a thistle tube and a delivery tube as in figure 67. Prepare to collect 3 bottles of gas over water. Pour in enough water to cover the copper and the lower end of the thistle tube and then add about half as much concentrated nitric acid. The action may be a bit slow in starting; if necessary, add from time to time small quantities of the concentrated nitric acid. If the action becomes very vigorous, add cold water.

After the air in the generator has been swept out, fill two bottles with the gas, nitric oxide (NO), and fill the third bottle half full. Let them stand in the trough for later use.

Note the color of the gas in the generating bottle just after the action starts, and again later. Also observe the color of the liquid left in the generator. This color is characteristic of the cupric ion (Cu^{++}).

Copy in your notebook and complete the equation:

$$3\,Cu + 8\,HNO_3 \longrightarrow \_\_\,Cu(NO_3)_2 + \_\_\_\_\_\_ \uparrow + \_\_\_\_\_\_$$

Properties. **1.** Let one bottle of nitric oxide escape into the air. *What evidence of chemical change do you observe?*

2. Cover a second bottle of the gas with a glass plate and set it upright on the table. Plunge a lighted taper into the gas, withdraw it at once, and replace the plate. *Does nitric oxide support the combustion of a taper? Does nitric oxide support the burning of phosphorus?* Line a deflagrating spoon with asbestos paper and put into it a bit of red phosphorus. Ignite the phosphorus and when it is burning vigorously plunge it into the same bottle.

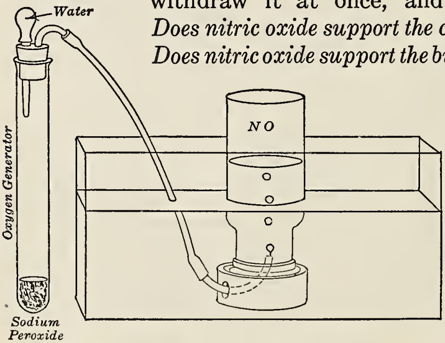

Fig. 68. Generating oxygen and introducing the gas into nitric oxide.

3. To study the effect of oxygen on nitric oxide, set up a little oxygen generator as in figure 68. Place in the large test tube about 2 cc. of sodium peroxide (Na_2O_2) and fill the dropper with water.* Immerse the delivery tube in the trough and let the water drop a little at a time on the peroxide. When the air has been displaced from the test tube, allow the oxygen to ascend slowly into the bottle which is only half full of nitric oxide. *Note the change in color. Does the volume of the gas change? If so, in which direction? Compare the result with that obtained by allowing air to mix with nitric oxide. With what gas in the air does the nitric oxide combine to become brown?* The brown gas is nitric dioxide (NO_2).

Summarize in parallel columns the properties of nitric oxide and nitric dioxide.

EXPERIMENT 43

Nitrous Oxide

Apparatus. — Small flask with 1-hole stopper. Large test tube with 2-hole stopper. Ring stand and clamp. Pneumatic trough. 4 wide-mouth bottles and glass squares. Test tube with 1-hole stopper. Glass tubing.

Materials. — Ammonium nitrate. Wood splint. Copper turnings. Concentrated nitric acid. Anhydrous copper sulfate.

Introduction. The gas which the dentist uses as an anesthetic is nitrous oxide (N_2O). It is conveniently prepared in the laboratory by heating ammonium nitrate (NH_4NO_3). This decomposes into water and nitrous oxide. Since all nitrogen compounds are unstable, it is particularly necessary to carry on this experiment with great care in order that the process may not proceed with explosive violence.

Preparation. Set up the apparatus as shown in figure 69. Put about 15 grams of pure crystallized ammonium nitrate (NH_4NO_3) into the flask and clamp it with the neck inclined slightly downward. The ammonium nitrate on heating decomposes into water and a gas, nitrous oxide (N_2O). Hence a condensing trap is inserted in the delivery tube. It is better to use warm water in the bottles and trough when collecting this gas.

---

* The instructor may have several of these oxygen generators set up and ready for use by the class.

The heat must be carefully regulated or an explosion may occur. Therefore it is advisable that two pupils work together on this experiment and that one of them give his *entire* attention to heating the flask gently and steadily.

Heat the nitrate gently until it is melted and a good deal of water of crystallization has been driven off. Keep the flame moving since overheating in one spot will cause trouble.

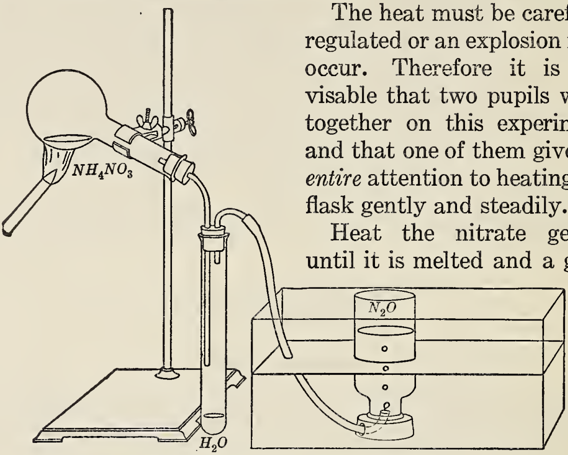

Fig. 69. Generating nitrous oxide.

Caution. If brown fumes appear in the flask during the heating, allow the flask to cool a little.

Let the first jarful of gas escape as it is mostly air from the flask and test tube. Collect three and a half bottles of the gas and then remove the delivery tube from the water before the heating is discontinued.

Identify the liquid in the large test tube by adding a little anhydrous copper sulfate. *Complete in your notebook the equation:*

$$NH_4NO_3 \longrightarrow N_2O \uparrow + \text{----}$$

Properties. **1.** *Describe the color and odor of the gas.*

2. Pour a little cold water into a bottle of the gas and cover it tightly with the palm of your hand. Shake the contents of the bottle vigorously. *What does this experiment show about the solubility of the gas in cold water?*

3. Plunge a glowing wood splint into another bottle of the gas. *What other gas have we already studied which behaves in much the same way?*

4. Prepare some nitric oxide by putting a few pieces of copper turnings into a test tube which is fitted with a stopper and a delivery tube. Cover the copper with water, add an equal volume of concentrated nitric acid, and at once insert the stopper. Let the nitric oxide bubble into water until the colored gas in the test-tube generator has disappeared. Then place the end of the delivery tube under the bottle which is half full of nitrous oxide. *Does the nitrous oxide react on nitric oxide in the same way as oxygen in Experiment 42?*

Questions. (1) *Why is it better to collect nitrous oxide over warm water?* (2) *Why does nitrous oxide support the combustion of a burning splint while nitric oxide does not?* (3) *How would you distinguish between nitric oxide and nitrous oxide?* (4) *Between nitrous oxide and oxygen?*

EXPERIMENT 44

Bromine — Preparation and Properties

Apparatus. — Ring stand and clamp. Test tubes and rack. 1-hole stopper to fit test tube. Wide-mouth bottle. Bunsen burner. Glass tubing.

Materials. — Sodium bromide. Manganese dioxide. Concentrated sulfuric acid. Carbon bisulfide. Chlorine water (saturate water with the gas).

Introduction. Bromine and iodine belong to the halogen family and therefore have much in common with chlorine, the chemical changes of which we have already studied (Exp. 15). Bromine can be prepared in much the same way as chlorine. In the laboratory we use a mixture of manganese dioxide, sodium bromide, and sulfuric acid. The equation is similar to that for making chlorine:

$$2\,NaBr + 2\,H_2SO_4 + MnO_2 \longrightarrow Na_2SO_4 + 2\,H_2O + Br_2 \uparrow$$

Bromine, however, is a liquid which has a suffocating odor. In fact, the name bromine means a "stench." Since it attacks the nose and throat even more violently than chlorine and also irritates the eyes and burns the skin, great care must be exercised in handling bromine in the laboratory.

Caution. **Always use the hood in preparing bromine.**

Iodine, on the other hand, is a solid which is prepared in exactly the same way as bromine and chlorine, namely, by warming a mixture of manganese dioxide and sodium iodide in sulfuric acid. Iodine is very easily vaporized and the vapor on cooling passes directly to a solid without forming a liquid. This process is called **sublimation** and will be used in Experiment 45 in preparing iodine.

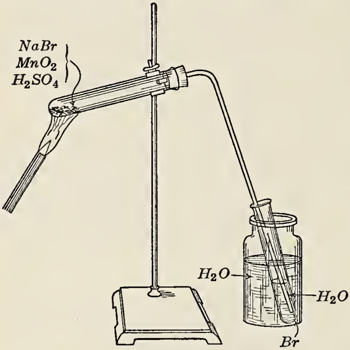

Fig. 70. Preparation of bromine.

Preparation.* Fit a cork stopper to a test tube and cut a hole through it. Insert a delivery tube and set up the apparatus as in figure 70. Mix on a piece of paper about 1 cc. of sodium bromide crystals with about the same bulk of manganese dioxide and put the mixture into the test tube. Add about 2 cc. of concentrated sulfuric acid to 1 cc. of water in a test tube and cool it. Then pour the acid on the mixture of sodium bromide and manganese dioxide. Insert the stopper carrying a delivery tube which extends to the bottom of the test tube containing about 10 cc. of water. It is well to stand the test tube in a bottle of cold water.

Heat the mixture very gently but not enough to boil the black liquid through the delivery tube. Stop heating when all the red vapor has been driven over, and at the same time remove the delivery tube from the water. *Why?*

---

* The instructor may prefer to do this experiment as a class experiment and use the glass retort as in making nitric acid.

Properties. 1. *Note the color of bromine vapor and the color of the liquid bromine.*

Fill the test tube containing the bromine about half full of water. Set the mixture aside.

Is bromine heavier or lighter than water? Is it soluble in water?

2. Pour about 3 cc. of the "bromine water" just prepared into a test tube and add not more than 1 cc. of carbon bisulfide. Cover the tube with your thumb and shake the contents vigorously. Most of the carbon bisulfide settles to the bottom. *Compare the colors of the bisulfide and water and so the relative solubility of bromine in these two liquids.*

Test for a bromide. Dissolve a small crystal of sodium bromide in about 5 cc. of water. *Note the color of the solution.* Add 2 cc. of carbon bisulfide and shake the mixture. *Why is the carbon bisulfide not colored by the bromine?*

Now add a few drops of freshly prepared chlorine water* and shake the contents vigorously. Allow the mixture to settle. *What substance has been set free by the chlorine? Complete the equation:*

$$\text{-- KBr} + Cl_2 \longrightarrow \text{----} + \text{----}$$

Summarize the properties of bromine: *color, odor, density of vapor* (compared with air), *density of liquid* (compared with water), *relative solubility in water and carbon bisulfide,* and *activity compared with chlorine.*

---

*A convenient way of making chlorine water is as follows: Drop a few small crystals of potassium chlorate into a test tube and cover them with concentrated hydrochloric acid diluted with an equal volume of water. Warm the tube a little and as soon as the chlorine begins to escape from the tube, fill it nearly full of water.

EXPERIMENT 45

Iodine — Preparation and Properties

Apparatus. — Test tubes and rack. Bunsen burner. Evaporating dish. Tripod. Crystallizing dish. Asbestos gauze. Mortar and pestle.

Materials. — Sodium iodide. Manganese dioxide. Concentrated sulfuric acid. Alcohol. Sodium iodide solution. Carbon bisulfide. Chlorine water. Bromine water. Sodium thiosulfate solution.

Preparation. Pour into an evaporating dish about 3 cc. of water and add about twice that volume of concentrated sulfuric acid. Pulverize several crystals (about 2 g.) of sodium iodide with about as much manganese dioxide and add the mixture to the acid. Set the evaporating dish on the asbestos gauze supported on the tripod and place a crystallizing dish half full of cold water upon it in such a way that the iodine vapor may be condensed on the bottom of the cool dish (Fig. 71).

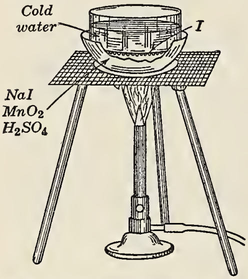

Fig. 71. Preparation of iodine.

Warm the evaporating dish with a very small flame. If the iodine vapor (violet) escapes, decrease the heat and refill the crystallizing dish with cold water. The iodine vapor condenses as a solid (*i.e.* sublimes) on the bottom of the cold dish. Scrape off the iodine crystals on a paper.

Complete and balance the following equation in your notebook:

$$\_\_\,NaI + \_\_\,H_2SO_4 + MnO_2 \longrightarrow Na_2SO_4 + MnSO_4 + \_\_\_\_ + \_\_\_\_\uparrow$$

Properties. *Solvents.* Try to dissolve a crystal of iodine in water by shaking it with a few cubic centimeters of water. *Is the iodine quite insoluble in water?*

To the water and iodine in the test tube add an equal volume of carbon bisulfide (or carbon tetrachloride) and shake the mixture. *Compare the solubility of iodine in water and in carbon bisulfide.*

Try the solubility of iodine in ethyl alcohol; also in solutions of sodium iodide and sodium thiosulfate ("hypo"). *Tabulate in your notebook the solubilities of iodine in these various solvents and the colors of the solutions as follows:*

SOLVENT	COLOR OF SOLUTION	SOLUBILITY (Very great, moderate, or slight)
Water		
Carbon bisulfide, etc. .		
Alcohol		
Sodium iodide sol. . .		

Replacement of iodine. Dissolve a small crystal of sodium iodide in about 5 cc. of water and then add a few cubic centimeters of carbon bisulfide. Shake the mixture. *Why is the liquid not colored with the iodine?*

Add a few drops of chlorine water and shake the liquids vigorously. Allow the mixture to settle. *What substance has been set free by the chlorine? Complete in your notebook the equation:*

$$\_\_\,NaI + Cl_2 \longrightarrow \_\_\_\_ + \_\_\_\_$$

To another portion of sodium iodide solution, add bromine water and carbon bisulfide. After shaking the mixture let it settle. *What substance has been set free by the bromine? Complete in your notebook the equation:*

$$\_\_\,NaI + Br_2 \longrightarrow \_\_\_\_ + \_\_\_\_$$

Assume that the more active element replaces the less active element; which of these halogens is the most active and which is the least active?

Summarize the properties of iodine: *color of solid, color of vapor, density of vapor* (compared with air), *density of solid* (compared with water), and *relative solubilities in the various solvents used.*

EXPERIMENT 46

The Halogen Acids

Apparatus. — Test tubes and rack. Bunsen burner.

Materials. — Sodium chloride. Sodium bromide. Sodium iodide. Concentrated sulfuric acid. Blue litmus paper. Concentrated ammonium hydroxide. Filter papers.

Hydrogen chloride. Put about 1 g. of common salt (sodium chloride, $NaCl$) in a test tube and add a few drops of concentrated sulfuric acid. If no action is visible, add a few drops more of sulfuric acid and warm the tube gently. *What happens?*

Blow across the mouth of the tube. *What is the result?* Hold a strip of moistened blue litmus paper near the mouth of the tube. *Result?* Hold a piece of filter paper which has been moistened in ammonia water near the tube. *Explain the formation of a white cloud.*

Write in your notebook the equation for the preparation of hydrogen chloride.

Hydrogen bromide. Repeat in every detail the experiment, using sodium bromide. *Answer the same questions.* Remember that all the hydrogen halides are colorless. *How do you account for the colored gas in the test tube? What element gives this color? What does it indicate as to the stability of hydrogen bromide? What gas is formed besides hydrogen bromide?* (Smell the gas very cautiously.)

Complete in your notebook the equations:

$$\text{--}\ NaBr + H_2SO_4 \longrightarrow Na_2SO_4 + \text{----}$$

$$H_2SO_4 + 2\ HBr \longrightarrow H_2SO_3 + \text{----} + \text{----}$$

$$\longrightarrow \text{----} + SO_2 \uparrow \text{----} + \text{----}$$

Hydrogen iodide. Again repeat the experiment as before, using sodium iodide. *Answer the same questions.* The amount of the fuming when you blow across the mouth of the test tube indicates the quantity of hydrogen iodide coming from the tube.

What is the colored vapor?

What gas, which you can recognize by its odor, is formed besides hydrogen iodide?

Complete in your notebook the equations:

$$8\,NaI + \_\_\,H_2SO_4 \longrightarrow \_\_\,Na_2SO_4 + \_\_\_\_$$
$$H_2SO_4 + 8\,HI \longrightarrow H_2S + \_\_\_\_ + \_\_\_\_$$

Comparison. *What does this experiment show with regard to the relative stabilities of hydrogen chloride, hydrogen bromide, and hydrogen iodide?*

Why are hydrobromic and hydriodic acids not usually prepared in this way?

EXPERIMENT 47

Sodium Carbonate and Bicarbonate

Apparatus. — Test tubes. 1-hole stopper to fit test tube. Ring stand and clamp. Bunsen burner. Beakers. Tripod and asbestos gauze. Glass tubing. Horn-pan balance and weights.

Materials. — Sodium bicarbonate (baking soda). Limewater. Potassium bitartrate (cream of tartar).

Effect of heating the bicarbonate. Put about 2–3 g. of sodium bicarbonate ($NaHCO_3$) into a test tube and clamp it so that the mouth is inclined slightly upward. Arrange the delivery tube so that it leads into a test tube containing limewater ($Ca(OH)_2$) (Fig. 72). Warm the bicarbonate gently with a small flame, taking care not to heat the test tube hot enough to color the flame yellow.

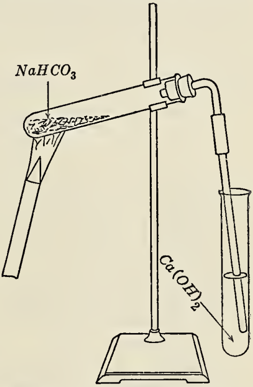

Fig. 72. Heating sodium bicarbonate and passing the gas into limewater.

What gas is evolved? What collects on the cool part of the test tube? To a part of the residue (Na_2CO_3) left in the tube add an acid. *What is the result?*

Complete in your notebook the equation for the action of heat on sodium bicarbonate:

$$\_\_\ NaHCO_3 \longrightarrow Na_2CO_3 + \_\_\_\_ + \_\_\_\_$$

Compare the taste of the bicarbonate with that of the carbonate.

Baking powder. Weigh out 2 g. of potassium bitartrate (cream of tartar, $KHC_4H_4O_6$). Calculate the amount of sodium bicarbonate required from the following equation:

$$\underset{\substack{\text{cream of}\\ \text{tartar}}}{KHC_4H_4O_6} + \underset{\substack{\text{baking}\\ \text{soda}}}{NaHCO_3} \longrightarrow \underset{\substack{\text{Rochelle}\\ \text{salt}}}{KNaC_4H_4O_6} + H_2O + CO_2 \uparrow$$

Weigh out this amount of sodium bicarbonate and mix the two salts thoroughly on paper.

Put half of this mixture into a beaker half full of *cold* water and the other half into another beaker half full of *hot* water. *What is the gas which escapes? What is the difference between the action of cold and of hot water on this mixture? Why is water needed in this chemical reaction?*

EXPERIMENT 48

Analysis of Baking Powder

(*Optional Experiment*)

Apparatus. — Test tubes and rack. Bunsen burner. Evaporating dish. Funnel. Beaker (small). Stirring rod. Ring stand and asbestos gauze.

Materials. — Sample of baking powder (2 tablespoonfuls, from home). Iodine solution. Barium chloride solution. Dilute hydrochloric acid. Concentrated sulfuric acid. Concentrated nitric acid. Ammonium molybdate solution. Sodium hydroxide solution. Filter papers.

Introduction. Sodium bicarbonate is an essential ingredient of all baking powders. *How could you prove its presence?* The other necessary ingredient is some solid substance which is weakly acidic, such as cream of tartar, calcium acid phosphate, or alum. A small amount of starch or flour is usually added to keep the mixture dry. The different kinds of baking powder vary according to the kind of acid agent present. Some powders have a mixture of two or even of all three of these acid-forming ingredients.

Directions. a. Place about 10 g. of a baking powder in a beaker and add about 50 cc. of water. Stir the mixture well until all the gas is evolved. Then filter and test the filtrate and residue for the various ingredients as follows:

b. *Starch. Will the starch be found in the filtrate or in the residue?* Test for starch with one drop of iodine solution as in Exp. 70.

c. *Sulfates.* Ordinary **alum** is a double salt of aluminum sulfate and potassium sulfate ($K_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24\ H_2O$). **Ammonium alum** is also a double salt of ammonium sulfate and aluminum sulfate ($(NH_4)_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24H_2O$). Thus it will be seen that if a baking powder contains alum, the filtrate will contain sulfates. Test the filtrate with barium chloride solution and dilute hydrochloric acid as in Exp. 27.

d. *Tartrates.* Pour about 5 cc. of the filtrate from (a) into an evaporating dish and add 5 drops of concentrated sulfuric acid. Evaporate to dryness over a small flame. Charring and the odor of burnt sugar indicate the presence of a tartrate.

e. *Phosphates.* If the baking powder contains calcium acid phosphate, it will be found in the filtrate from (a). To test for a phosphate, take a few cubic centimeters of the filtrate and acidify with a few drops of nitric acid. Warm the mixture and add a few drops to 5 cc. of ammonium molybdate solution ($(NH_4)_2MoO_4$). Set it aside. A bright-yellow precipitate indicates a phosphate.

f. *Ammonium salts.* If the baking powder contains ammonium alum, we can detect it by pouring about 5 cc. of the filtrate into a test tube and adding the same volume of sodium hydroxide solution. If it is heated to boiling, ammonia gas is evolved.

g. *Calcium and aluminum.* Since a baking powder containing sulfates always contains aluminum and one containing phosphates always contains calcium, we need not make special tests for these metals here.

Results. In making the report of your chemical analysis of baking powder, state *where you obtained it, the name of the manufacturer, exact name of the brand, what you tested for and how, and what you found present.*

EXPERIMENT 49

Hydrolysis of Salts

Apparatus. — Test tubes and rack. Stirring rod.

Materials. — Litmus papers (red and blue). Copper sulfate, aluminum sulfate, ferric chloride, sodium chloride, potassium nitrate, sodium carbonate, and sodium tetraborate (borax).

Introduction. Not all salt solutions react neutral when tested with litmus paper. This is due to the fact that water is itself dissociated very slightly into H^+ and OH^-. The action of the ions of water on a dissolved salt is called **hydrolysis.** When the ions of the salt unite with the ions of water to form the undissociated molecules of a weak base, an equivalent amount of hydrogen ions (H^+) is set free and the solution gives an **acid reaction** with litmus. When the ions of the salt unite with the ions of water to form the undissociated molecules of a weak acid, an equivalent amount of hydroxyl ions (OH^-) is set free and the solution gives a **basic reaction** with litmus.

NOTE. Remember that the reddening of blue litmus indicates H^+ ions and the turning blue of red litmus, OH^- ions.

Directions. *Testing salt solutions with litmus.* Dissolve about 0.5 g. of each of the following substances in 10 cc. of distilled water in separate test tubes: copper sulfate ($CuSO_4$), sodium chloride ($NaCl$), sodium carbonate (Na_2CO_3), aluminum sulfate ($Al_2(SO_4)_3$), ferric chloride ($FeCl_3$), potassium nitrate (KNO_3), and sodium tetraborate ($Na_2B_4O_7$).

Test each solution with litmus paper by putting a small drop of the liquid on the paper with a clean glass rod. (Be sure to rinse the rod for each new salt solution.)

It will be convenient to record the results in your notebook in a tabular form as follows:

Salt	Formula	Action with Red Litmus	Action with Blue Litmus
Copper sulfate . . .			
Sodium chloride . .			
Sodium carbonate .			
Aluminum sulfate . .			
Ferric chloride . . .			
Potassium nitrate . .			
Sodium tetraborate .			

Results. *Divide these solutions into three classes:* (1) those that give an *acid* reaction, (2) those that give a *basic* reaction, and (3) those that are *neutral.* Take one case from each of these three classes and *explain in terms of the ionic theory the reaction of the solution toward litmus. Write in your notebook equations to illustrate hydrolysis in classes 1 and 2.*

EXPERIMENT 50

Flame Tests

Apparatus. — Bunsen burner. Platinum (or nichrome) wire mounted in a glass tube. Test tubes. Cobalt glass (2 or 3 squares).

Materials. — Concentrated hydrochloric acid. Chlorides of sodium, potassium, lithium, calcium, strontium, and barium. Unknowns.

Introduction. There are certain metals which are easily detected in whatever salt they may occur because of a characteristic color which each imparts to the Bunsen flame. This furnishes a convenient and often a very delicate qualitative test for these metals. We shall study compounds of **sodium, potassium, lithium, calcium, strontium,** and **barium.** We may use a piece of platinum wire which has a little hook bent at one end and is melted into a short piece of glass tube at the other end. (Nichrome wire may be substituted here, and one end may be twisted around a match stick to serve as a handle.)

Hold the end of the platinum wire in the blue Bunsen flame. If the flame is colored by it, dip the wire in a little concentrated hydrochloric acid in a test tube and again hold it in the flame. Repeat this several times until the wire ceases to impart any color to the flame.

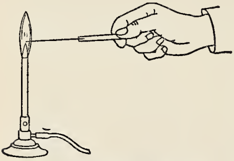

Fig. 73. Flame test for sodium, etc.

Directions. a. *Sodium.* Heat the end of the cleaned wire and quickly touch a tiny particle of sodium chloride. Hold the wire with the adhering particle in the lower part of the outer blue layer of the flame (Fig. 73) and *note the color of the flame produced.* Examine the sodium flame through two thicknesses of blue (cobalt) glass. *What tint of light is absorbed by the blue glass and hence does not reach the eye?* Hold the wire in the flame until the color has nearly disappeared; then clean it as before by dipping in hydrochloric acid.

b. *Potassium.* When the wire is quite clean, heat it and touch a bit of potassium chloride. *Record the color imparted to the flame.* Hold two thicknesses of blue glass between the flame and the eye and observe whether the color is absorbed.

Mix together intimately a little sodium chloride and potassium chloride and plunge the hot wire into the mixture. *Record the color imparted to the flame. Why do you not see both of the characteristic colors?* Now examine the flame of the mixture through blue glass. *Which metal can you recognize in this way?*

c. *Lithium, calcium, strontium, and barium.* When the wire is thoroughly clean and imparts no color to the flame, plunge it into a little lithium chloride and hold it in the flame. *Note the characteristic color.*

Repeat this experiment with the chlorides of the other three metals (Ca, Sr, and Ba), and tabulate your results:

Substance	Formula	Color of Flame	Remarks

d. *Test for unknown.* Obtain from the instructor a numbered envelope containing an "unknown." This may contain one or two salts of these six metals. *Record the number of your unknown and identify it.*

EXPERIMENT 51

Acids of Phosphorus

Apparatus. — Wide-mouth bottle. Glass square. Deflagrating spoon. Beaker. Tripod. Asbestos gauze. Bunsen burner. Test tubes and rack. Forceps.

Materials. — Yellow phosphorus. Dilute nitric acid. Dilute ammonium hydroxide. Silver nitrate solution. Ammonium molybdate solution. Litmus papers.

Caution. Yellow phosphorus takes fire readily. It should always be handled with forceps.

Preparation. Place about 20 cc. of distilled water in a clean wide-mouth bottle or pint fruit jar. Line a deflagrating spoon with asbestos paper and put in it a piece of yellow phosphorus. Ignite it by touching it with a hot wire and lower the burning phosphorus into the bottle or jar, quickly covering the mouth of the bottle with a glass plate (Fig. 74). As soon as the phosphorus stops burning, remove the spoon and shake the bottle to dissolve the phosphorus pentoxide (P_2O_5).

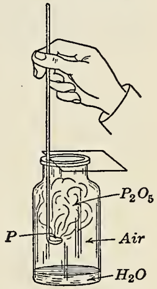

Fig. 74. Burning phosphorus on a deflagrating spoon in a bottle with a little water.

Test the solution with litmus paper. Write in your notebook the equations for (*a*) *burning phosphorus in air and* (*b*) *dissolving phosphorus pentoxide in water.*

Pour 3 cc. of dilute nitric acid into the solution in a beaker and boil it down to about half its original volume. The hydrogen ion of the nitric acid acts as a catalyst to convert the metaphosphoric acid (HPO_3) by addition of water to orthophosphoric acid (H_3PO_4). *Write the equation.*

Tests. Add to a portion of the orthophosphoric acid nearly enough dilute ammonium hydroxide to neutralize the acid; then add silver nitrate solution drop by drop until a precipitate is formed. *Name and describe this precipitate. Write the equation.*

To about 10 cc. of a solution of ammonium molybdate add a few drops of the orthophosphoric acid. Let the mixture stand for several minutes and then warm gently. A precipitate shows the presence of the phosphate ion (PO_4^{---}). *Describe the precipitate.*

Try the effect of adding ammonium hydroxide solution until the solution is alkaline. *Note the result.* Again acidify with nitric acid and *record the result.*

EXPERIMENT 52

Soil Testing

(*Optional Experiment*)

Apparatus. — Beaker. 3 pint fruit jars. Graduated cylinder. Magnifying glass.

Material. — Sample of soil (from home garden). Litmus papers.

Litmus test for acidity. Place some soil in a beaker or tumbler and moisten it well with distilled water. Put a strip of blue litmus paper on the moist mass. If the soil is acid, the litmus paper will turn pink within 15 minutes.

Sometimes it is necessary to test soil for acidity where distilled water is not available. In this case the test may be made by taking a handful of moist soil direct from the field and pressing it into a ball. Then break the ball in two so that the litmus paper may be placed between the two halves. After 10 minutes examine the litmus paper.

NOTE. It is well to handle the litmus paper with forceps because the perspiration of the hands is usually slightly acid. Use only the best litmus paper.

What are the means of correcting acidity in the soil?

Percentage of sand, silt, and clay. Put about 2 tablespoonfuls of fine soil in a pint fruit jar three-fourths full of water. Shake the mixture vigorously for several minutes and then let it stand for one minute. Pour off the muddy water into a second jar. The sediment remaining in the first jar is composed mainly of *sand.*

When the second jar has been standing 5 minutes, pour off the muddy water into a third jar. Add more water to this jar and shake it vigorously. After it has stood for at least 2–3 hours, pour off the water. The residue left in the second jar was largely *silt;* the residue in the third jar is almost entirely *clay.* Dry each of these three residues and determine the volumes of each portion in a graduated cylinder. *Compute the percentage of sand, of silt, and of clay in the soil. Note how fine the particles of each ingredient are and examine them under a magnifying glass.*

EXPERIMENT 53

A Fusible Alloy

Apparatus. — Iron crucible, 20 cc. Pipe-stem triangle. Forceps. Bunsen burner (or Fisher form of Méker burner). Ring stand. Cover of pasteboard box. Iron wire, 6″ of # 14. Evaporating dish. Horn-pan balance and weights.

Materials. — Bismuth, gran. Cadmium, mossy. Lead, gran. Tin, gran.

Introduction. When two or more metals are melted together and the resulting product is allowed to solidify, the product is called an **alloy**. Although alloys have been known since ancient times, it is only recently that they have assumed an enormous importance both commercially and theoretically. Among the alloys which are easily fusible and so can be prepared in the laboratory is Wood's metal. This is composed of bismuth 50%, lead 25%, tin 13%, and cadmium 13%; it melts at about 60° C. Its low melting point makes it very useful for plugs in automatic fire-sprinkler systems. In general, an alloy has a lower melting point than its component metals. In other words, the melting point of a pure metal can be lowered by adding another metal.

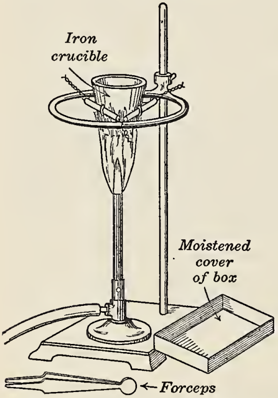

Fig. 75. Melting metals in an iron crucible.

Directions. a. ***Preparation.*** Use an iron crucible to melt the component metals and support it in a pipe-stem triangle on the ring of the iron support stand, as shown in figure 75. Use a Bunsen burner or, if available, the Fisher form of the Méker burner. While the crucible is getting hot, weigh out about 10 grams of bismuth, 5 grams of lead, 2.5 grams of tin, and 2.5 grams of cadmium. Mix on a sheet of paper the lead and tin, and place in the crucible. When these are melted, add the bismuth and cadmium. Stir with an iron wire until the whole mass is thoroughly liquefied.

Moisten the cover of a small pasteboard box and tilt it against the base of the iron stand. With forceps lift the crucible from the flame and carefully pour the molten alloy into the lower edge of the box cover. This liquid may be solidified by pouring a little water on it.

b. *Properties. Examine the alloy and compare its appearance with that of the component metals.*

Determine its density (g./cc.) by weighing a sample on your horn-pan scales and by measuring its volume by putting it in a graduated cylinder partly filled with water.

Heat some water in an evaporating dish until it boils. Then put in a piece of the alloy which you have prepared. *What happens to the alloy?*

The melting points of the components are as follows: bismuth 269° C., cadmium 321° C., lead 327° C., and tin 232° C.

How does the melting point of the alloy compare with the melting points of its components?

EXPERIMENT 54

Solutions of Gases, Liquids, and Solids

Apparatus. — Test tubes and rack. Bunsen burner. Funnel. Watch glasses.

Materials. — Concentrated ammonium hydroxide. Carbon tetrachloride. Glycerol. Copper sulfate. Potassium chlorate. Calcium carbonate (powdered). Filter papers.

Introduction. Water is our most important solvent not only for solids but for liquids and gases as well. When a solid has been mixed with water, it is impossible to see the separate particles, even with a powerful microscope. The solid has been subdivided into very small particles. Therefore a solution offers a large surface on which chemical action may take place.

A very large number of the important chemical changes in nature and in the industries are brought about between substances in solutions. Some substances are said to be insoluble in water, but this is not strictly true since even such a substance as glass dissolves to a very slight extent in water. In this experiment and the next you will learn that the solubility of various substances depends upon several factors.

Gases in water. Warm gently in a test tube a little water drawn from the faucet. *Is there any evidence that a gas has been dissolved in the water?*

In the same way heat gently a little ammonia water (ammonium hydroxide) and note any evidence that a gas has been dissolved in the water.

Are gases more soluble in hot or in cold water?

Liquids in liquids. Add to 5 cc. of water in a test tube 5 cc. of carbon tetrachloride, a few drops at a time, and shake the mixture vigorously after each addition. *Are water and carbon tetrachloride miscible at each stage?*

Repeat, using glycerol and water.

Solids in liquids. Place in separate test tubes about 1 g. of each of the following powdered substances: copper sulfate ($CuSO_4$), potassium chlorate ($KClO_3$), and calcium carbonate ($CaCO_3$). Add to each substance 10 cc. of water and shake each test tube.

What evidence is there that these substances have dissolved?

If there is any doubt as to whether any of a solid has dissolved, filter off 5 cc. of the liquid and evaporate on a watch glass to dryness.

If in any case all the solid has dissolved, add another gram of the powdered solid and continue this process as long as it is completely dissolved.

What are the relative solubilities of these three substances in water?

EXPERIMENT 55

Effect of Temperature on Solubility

Apparatus. — Test tubes and rack. Ring stand. Bunsen burner. Asbestos gauze Large beaker.
Materials. — Copper sulfate. Sodium chloride.

Solubility in cold water. Add to 10 cc. of water in a test tube 2 g. of well pulverized copper sulfate and shake the tube until the solid dissolves. Continue adding 2-gram portions until the solution is saturated, that is, until some of the solid remains undissolved after shaking. *Calculate the amount* (g.) *which would dissolve in* 100 *g. of cold water.*

Repeat the experiment, using sodium chloride (common salt) instead of copper sulfate.

Solubility in hot water. Heat the solutions just prepared in a beaker of boiling water (Fig. 76) and continue to add 2-gram portions of the solid until each solution is saturated at 100° C. *Compute for each solid the amount which dissolves in* 100 *g. of hot water.*

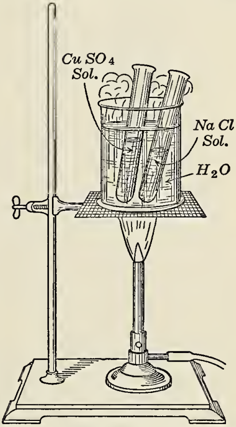

Fig. 76. Heating solutions in hot water.

Cooling a saturated solution. Cool the solutions just prepared by holding the test tubes in running water from the faucet. *Observe the amount and size of the crystals formed in each tube.*

Warm the contents of the tubes carefully by putting them once more into the boiling water until the crystals are again dissolved. Set the tubes away in the test-tube rack and let them cool slowly until the next laboratory period. *Compare the crystals formed quietly and slowly with those formed quickly.*

EXPERIMENT 56

Solubility of Common Salt

(Quantitative Experiment)

Apparatus. — Small flask with stopper. Evaporating dish. Horn-pan balance and weights. Ring stand. Copper water bath or large beaker. Tripod. Bunsen burner. Thermometer. Funnel.

Materials. — Sodium chloride. Filter paper.

a. First make a saturated solution of sodium chloride. This can be done by placing 10 g. of common salt with 20 cc. of water in a stoppered flask or bottle and shaking the contents at intervals for 15 minutes; *or* by dissolving 10 g. of salt in 20 cc. of boiling water and allowing the solution to cool to the temperature of the room (shaking the solution occasionally). In either case some of the salt should remain undissolved.

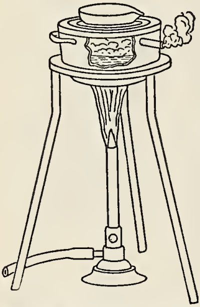

Fig. 77. Evaporating over a water bath (copper).

b. Take the temperature of the saturated salt solution just prepared. Carefully weigh a clean, dry evaporating dish and then pour into it through a filter a portion of the solution. Reweigh as rapidly as possible, but carefully.

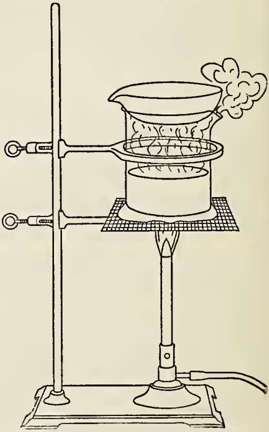

Fig. 78. Evaporating on a beaker of boiling water.

c. Evaporate the solution completely to dryness upon a water bath (Fig. 77) or on a beaker of boiling water (Fig. 78). Be careful not to lose any of the salt by spurting. Let the dish and contents cool and again weigh. Reheat the dish and contents until the weight is constant.

NOTE. Care must be taken to replace the water from time to time in the copper bath or beaker.

d. *Compute the number of grams of salt soluble in 100 g. of water at the observed temperature.* Compare your result with the results plotted in the curves shown in figure 209 on page 374 of the textbook. *Record in your notebook your data and results in tabular form somewhat as follows:*

Data:
Temperature of solution . . . . °C.
Wt. of dish + solution . . . . g.
Wt. of dish . . . . g.
Wt. of solution . . . . g.
Wt. of dish + dry solid . . . . g.
Wt. of dish . . . . g.
Wt. of salt . . . . g.
Calculations:
Wt. of water . . . . g.
Wt. of salt dissolved in 100 g. of water at __ °C. . . . . g.

EXPERIMENT 57

Water of Hydration — Efflorescence and Deliquescence

Apparatus. — Three watch glasses. Ring stand with clamp. Bunsen burner. Test tubes.

Materials. — Crystals of sodium carbonate and gypsum. Fused calcium chloride. Crystals of potassium chlorate, potassium nitrate, potassium alum, zinc sulfate, sodium sulfate, and copper sulfate.

Introduction. Some substances, such as washing soda, which are crystalline in form, crumble to a powdery mass when exposed to the air for a few days, and their crystalline form disappears. Careful observation shows that these crystals have given off water vapor. This water is an essential part of the crystalline form, and when it leaves the crystal, the form disappears. This process is called **efflorescence**. Crystals are usually formed from solutions, and the water is built into the crystal at the time of its formation. Such a crystal containing water of crystallization is called a **hydrate**. But the water is not so firmly combined as its other constituents, and very often a moderate heat will drive off this water of hydration. When all the water has been removed, the substance is said to be **dehydrated**, or in an anhydrous form. Certain substances, when exposed to the air, automatically take water from the air in sufficient quantity to make a solution of the substance. This process is called **deliquescence** and is particularly valuable in a drying agent.

Directions. **a.** Place a fresh glassy crystal of washing soda (sodium carbonate) on a watch glass and set it aside until the next laboratory period. Treat in the same way a small crystal of gypsum and a piece of fused calcium chloride. Be sure to label each watch glass. *After a day or two describe any changes which you observe in the appearance of each substance.*

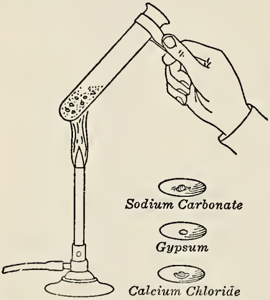

Fig. 79. Testing substances for water of crystallization.

b. Place a clear crystal of washing soda in a dry test tube and clamp the tube in a horizontal position. Heat the crystal gently and observe any changes in its appearance. *What condenses on the walls of the tube?*

c. In separate dry test tubes warm gently (Fig. 79) a few crystals of each of the following substances: potassium chlorate, potassium nitrate, potassium alum, zinc sulfate, and sodium sulfate.

Record in your notebook the results in tabular form as follows:

SUBSTANCE HEATED	AMOUNT OF CONDENSED WATER	APPEARANCE OF RESIDUE

NOTE. The amount of condensed water may be indicated by the words, "a trace," "little," or "much." Even anhydrous substances often contain a little moisture, which is said to be "water mechanically held."

Are all crystalline substances hydrates?

d. In another test tube heat gently a small crystal of copper sulfate (blue vitriol). *Observe the change in color and form.*

When the tube is cold, add a few drops of hot water. *Note the change in color.* Pour the solution on a watch glass and set it aside to recrystallize. *Compare the product with the original substance.*

EXPERIMENT 58

Fractional Crystallization *

Apparatus. — Ring stand or tripod. Asbestos square. Bunsen burner. 250-cc. beaker. Funnel. Test tubes and rack.

Materials. — Mixture of copper nitrate and potassium chlorate (1:1 by weight). Filter paper.

Introduction. Crystals are chemically homogeneous bodies having regular geometrical forms. The form of the crystals is characteristic of the substance. In the process of crystallization from solution, there is a tendency to exclude from the crystal all matter not homogeneous with it. Hence, if the process of crystallization is not too rapid, and if the solution is not too concentrated, this process is a valuable way of purifying salts.

In the process of fractional crystallization, the separation of the two substances depends on the relative solubilities of the substances in hot and cold solutions. In this experiment we shall use a 1:1 mixture of cupric nitrate and potassium chlorate. It so happens that these two compounds differ markedly in their solubilities at varying temperatures, as is shown in the table on the next page:

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* This experiment is based on a similar experiment used in Chemistry A in the Chemical Laboratories of Harvard University.

	20° C.	40° C.	60° C.	80° C.	100° C.
Copper nitrate	56 g.	62 g.	64 g.	68 g.	
Potassium chlorate . .	7 g.	14 g.	25 g.	39 g.	57 g.

It will be seen that copper nitrate is much more soluble in water than potassium chlorate, and potassium chlorate is nearly eight times as soluble in boiling water as in water at 20° C.

Directions. Add 30 grams of this mixture to 50 cc. of water in a 250-cc. beaker. Heat and at the same time stir the mixture until the solution boils. All the solid should be dissolved. Filter the hot solution as rapidly as possible (use a crinkled filter paper). Then concentrate the filtrate by evaporation until crystals of potassium chlorate begin to appear in the boiling solution. At this point stop heating and set the beaker aside to cool to room temperature. (If a light-greenish precipitate appears, this is a basic salt of copper, which can be readily dissolved by adding 2 cc. of dilute nitric acid to the solution.) When the solution is at room temperature, as determined by using a thermometer, filter and wash the crystals with 10 cc. of cold water. In this process let all the liquid drain from the filter paper before washing the crystals a second and a third time.

In case the potassium chlorate crystals are bluish, redissolve them in the minimum volume of boiling water and set the solution aside to crystallize. To dry the crystals, let them remain several hours on the opened filter paper.

Since potassium chlorate does not form hydrates and is neither deliquescent nor efflorescent, it is especially convenient to prepare. When pure, the product is white and dry. Weigh a small empty bottle, put in your potassium chlorate, and reweigh. These weighings need only be to 0.1 gram. Label the bottle with your name, the date, the experiment number, gross weight, tare weight, and net weight. Hand this to your instructor with your report.

EXPERIMENT 59

Colloids

(Class Experiment)

Apparatus. — Parchment paper (circles 10 cm. in diam.). Mortar and pestle. Beaker. Test tubes. Bunsen burner.

Materials. — Starch. Solutions of sodium chloride, silver nitrate, gelatin (2 per cent), sodium silicate (sp. gr. about 1.1), iodine, and soap. Mineral oil. Concentrated hydrochloric acid.

Introduction. In colloids the particles are much larger than simple molecules and yet so small that they will not settle out. Some substances, like starch, glue, and gelatin, always dissolve to give a colloid. Other substances with relatively simple molecules can under certain conditions often be obtained as colloids. It is possible to prepare membranes (for example, of collodion) which will allow salts and other substances to pass through but which will retain colloids. This process of separation is called **dialysis.** Small amounts of one colloidal substance will often prevent the precipitation of another relatively unstable colloid. There are many practical illustrations of the use of **protective colloids.** Casein acts as a protective colloid in milk, keeping a considerable amount of the butter fat in solution. Many colloids on cooling or on concentration by evaporation turn into jelly-like materials, which are called **gels.** In this experiment we have to do with these colloidal phenomena.

Directions. a. *Dialysis.* Rub a pinch of starch to a smooth paste with a few drops of cold water in a mortar. Add a little common salt mixed with distilled water. Make a dialyser by folding a circle of parchment paper (free from pinholes) into a cone as if to fit a funnel and fastening it with two paper clips at opposite sides of its circumference. Fill the dialyser two-thirds full of the starch-salt solution and *carefully* float it in a large beaker of distilled water (Fig. 80). Test small portions of the starch-salt solution for starch by adding a drop of iodine solution.* The iodine is absorbed by the colloidal starch particles and gives a blue color. The salt may be tested for by showing the presence of a chloride by means of silver nitrate solution (Exp. 17). From time to time test the surrounding distilled water for starch and a chloride. *Which of the two substances, starch or salt, passed through the membrane?*

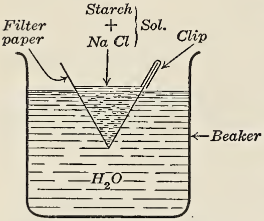

Fig. 80. Dialysis of starch through filter paper.

b. ***Protective colloids.*** To a few drops of silver nitrate solution, add an excess of sodium chloride. Shake the mixture vigorously, and note whether the precipitate settles out. Repeat, using a silver nitrate solution which has been mixed with its own volume of a gelatin solution (about 2 per cent gelatin). *What effect does the gelatin seem to have?*

Shake a little mineral oil (machine oil) with water. Then shake oil with a soap solution. *Compare the results and explain.*

c. ***Formation of gels.*** Concentrate a little gelatin solution to about a 10-per cent solution, and cool it under running water. If it gelatinizes, determine whether it will liquefy again on being immersed in a beaker of hot water. *Is this change reversible?*

To a little water-glass (sodium silicate) solution, add 2 or 3 drops (not more) of concentrated hydrochloric acid. If it does not immediately gelatinize, cool it and set it aside. *What is the gelatinized product* (p. 413)?

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* Iodine solution may be prepared by dissolving 2 g. of iodine and 10 g. of potassium iodide in 100 cc. of water.

EXPERIMENT 60

Treatment of Hard Water

Apparatus. — Carbon dioxide generator (Exp. 53). Large test tube. Test tubes and rack. 2 small beakers. Funnel. Bunsen burner.

Materials. — Limewater. Marble chips. Concentrated hydrochloric acid. Calcium sulfate solution. Magnesium sulfate solution. Sodium carbonate solution. Filter papers. Concentrated ammonium hydroxide. Ammonium oxalate solution. Ammonium chloride solution. Solution of disodium phosphate.

Introduction. Hard water is undesirable for industrial and household uses for two reasons. In the first place, when such water is used in the boiler, it deposits a heavy crust, called "boiler scale." This is not only a poor conductor of heat but also dangerous for the life of the boiler. In the second place, hard water is unsuitable for washing purposes because it will not easily form a lather with soap. The hardness of water is due to the mineral matter which is in solution. If these minerals in solution are chiefly calcium and magnesium bicarbonate, the water is said to have temporary hardness. If the minerals are sulfates of these same metals, the water is permanently hard. In this experiment we shall learn some of the methods of treatment for these two kinds of hard water.

Directions. a. *Temporary hardness.* Dilute about 10 cc. of saturated limewater by adding an equal volume of water. Pass a steady stream of carbon dioxide through the half-saturated limewater until the precipitate ($CaCO_3$) is formed and then is redissolved. The clear liquid contains calcium bicarbonate ($Ca(HCO_3)_2$) and is called water of temporary hardness. *Write two equations to represent these changes.*

b. *Removing temporary hardness.* To about 5 cc. of the hard water just prepared, add about 3 cc. of saturated limewater and shake the mixture. *What is the precipitate? Complete in your notebook the equation:*

$$Ca(HCO_3)_2 + Ca(OH)_2 \longrightarrow \text{------} \downarrow + \text{----}$$

Boil for several minutes about 5 cc. of the hard water prepared in (a). The acid salt (bicarbonate) is changed back into the normal salt with the loss of carbonic acid. Set the tube with contents aside to settle. *Complete in your notebook the equation:*

$$Ca(HCO_3)_2 \longrightarrow CaCO_3\downarrow + \text{----} + \text{----}$$

Compare the amounts of precipitated calcium carbonate formed by boiling and adding limewater. Explain.

c. ***Permanent hardness.*** Pour 10 cc. of a solution of calcium sulfate* into a small beaker and the same volume of magnesium sulfate solution into another beaker. Add to each 5 cc. of sodium carbonate solution. (Compare the solubilities of calcium carbonate and sulfate. Table in Appendix.) Filter each solution. *What is the precipitate in each case? Write the ionic equations for these reactions.*

Add to the filtrate from the calcium sulfate solution just prepared 5 cc. of concentrated ammonium hydroxide and 5 cc. of a solution of ammonium oxalate. If the calcium ion (Ca^{++}) is present, a white precipitate of calcium oxalate will be formed.

Add to the filtrate from the magnesium sulfate solution which has just been prepared 5 cc. of a solution of ammonium chloride, 5 cc. of concentrated ammonium hydroxide, and 5 cc. of a solution of disodium phosphate. If the magnesium ion (Mg^{++}) is present, a white precipitate of magnesium ammonium phosphate will be formed.

Results. *What does this experiment show in regard to "softening" permanently hard waters with sodium carbonate?*

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* The calcium sulfate solution should be prepared some time beforehand. Shake some plaster of Paris with water; let it stand for some time and then filter.

EXPERIMENT 61

Mortar and Concrete

Apparatus. — Ring stand with smallest ring. Iron wire (# 16). Méker burner. Evaporating dish. Test tubes. 2 wide-mouth bottles with corks.

Materials. — Marble chips. Lumps of quicklime. Litmus papers. Sand (clean, sharp). Dilute hydrochloric acid. Portland cement.

Introduction. Mortar is made by mixing sand with wet, freshly slaked lime. Lime is ordinarily made by heating limestone ($CaCO_3$) to a red heat. Cement is believed to be a mixture of calcium silicate and calcium aluminate. When this is mixed with water, sand, and broken stone, the product hardens and forms concrete, with which we are all familiar in buildings, roads, and dams. The advantage that cement has over mortar which has led to its very extensive use in recent years is that cement will harden even under water and can, therefore, be employed in the construction of dams, foundations of bridges, and similar structures. In this experiment we shall see how lime is prepared from limestone, and how it is used to make mortar. Then we shall use cement with sand to make concrete.

Directions. a. *Making quicklime.* Wrap an iron wire around a small chip of marble (about 5 mm.) and hang it in the hottest part of the flame of a Méker burner (Fig. 81) or blast lamp. Heat it as hot as possible for 15–20 minutes. The product is

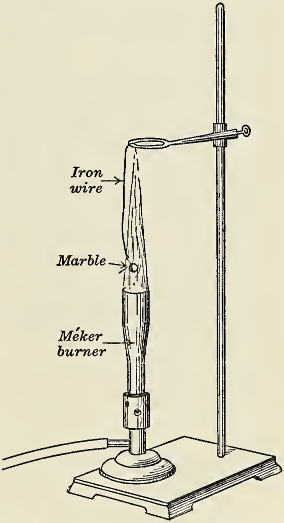

Fig. 81. Heating a chip of marble.

calcium oxide, or quicklime (CaO). *Compare it with marble. Write the equation for this reaction.*

b. *Slaking quicklime.* Pour a little boiling water on a lump of quicklime (about 25 g.) in an evaporating dish. Pour the water upon it a little at a time as long as it is taken up *readily.* Do not add too much water. Watch the lump for some time and note any changes (heat, volume, appearance). At the end you should have a smooth, stiff paste. *Test with litmus and write the equation for this reaction.*

c. *Mixing mortar.* Mix with the paste just prepared about 50 g. of sand and add more water if required. Make up part of the mortar into a ball and place in a wide-mouth bottle. Cork it tightly. Spread the rest on a board. After two days compare the two samples of mortar. Test each with dilute hydrochloric acid. *Explain the difference in the two samples of mortar. Write the equation for the setting of mortar.*

d. *Portland cement.* Mix a tablespoonful of cement with twice its bulk of clean sharp sand and then add slowly enough water to make a stiff paste. Take part of this mixture and make a small ball. Place this in a wide-mouth bottle and cork it tightly. Pour the rest of the paste into a small pasteboard or wooden box which has been greased. After two days examine each sample of concrete. *What advantages has concrete over mortar as a building material?*

EXPERIMENT 62

Compounds of Silicon

(Optional Experiment)

Apparatus. — Mortar and pestle. Iron crucible. Pipe-stem triangle. Tripod. Bunsen burner. Funnel. Evaporating dish. Asbestos gauze. Beaker (250 cc.).

Materials. — Clean sand (silica). Sodium carbonate. Sodium silicate (solution). Filter papers. Dilute hydrochloric acid. Crystals of copper sulfate, nickel sulfate, cobalt nitrate, zinc sulfate, and ferrous sulfate.

Sodium silicate. Thoroughly mix 1 g. of clean sand (SiO_2) with 5–6 g. of dry sodium carbonate by grinding them together in a mortar. Put the mixture into an iron crucible and heat gently over a Bunsen flame until the effervescence ceases, and then to a red heat until the whole mass is fused. Allow the fused mass to become thoroughly cold and then dissolve as much of it as is soluble in 25 cc. of boiling water. Filter the mixture and discard the residue. Concentrate the filtrate by evaporating the solution to about one-third its original volume. This solution contains sodium silicate, or water glass (Na_2SiO_3).

Silicic acid. Acidify the solution of sodium silicate by the addition of dilute hydrochloric acid. The gelatinous precipitate is silicic acid (H_2SiO_3). Slowly evaporate the acid mixture, heating it gently to avoid sputtering, until a dry, white powder remains. Heat almost to redness for a few minutes and then let it cool.

Extract the sodium chloride from the residue by adding 25 cc. of water and boiling gently for several minutes. Identify what remains.

What was formed during the fusion? Write the equation.

What was formed by adding hydrochloric acid? Write the equation.

What was the object of heating nearly to redness? Write the equation.

A chemical recreation. Mix thoroughly in a beaker about 30 cc. of the commercial solution of water glass with about twice its volume of water. Drop a small crystal of each of the following substances into this solution: copper sulfate, nickel sulfate, cobalt nitrate, zinc sulfate, and ferrous sulfate. Let the solution stand quietly over night. This is sometimes called "**The Chemist's Garden.**" Record and explain the changes observed.

EXPERIMENT 63

Acetylene and Methane

Apparatus. — Test tubes. Ignition tube with 1-hole stopper. Glass tubing. 250-cc. wide-mouth bottle. Ring stand and clamp. Evaporating dish.

Materials. — Calcium carbide. Limewater. Soda lime. Sodium acetate (powdered, anhydrous).

Introduction. Methane, or marsh gas, is formed in marshes and is produced by the decay of vegetable matter. It is often present in coal mines and forms a considerable part of natural gas. Ordinary coal gas contains from 35 to 40 per cent by volume of methane. Acetylene is another hydrocarbon which is valuable as an illuminant and as a fuel gas. While it is unsafe to compress acetylene in tanks, yet it may safely be dissolved under pressure in acetone. This is the way it is compressed in *Presto-Lite* cylinders. The oxyacetylene blowpipe gives an exceedingly hot flame much used in cutting and welding metals.

Directions. **1.** *Acetylene* (H_2C_2). Invert a test tube filled with water in an evaporating dish half filled with water. Drop a piece of calcium carbide (CaC_2), the size of a pea, into the water and quickly place the mouth of the test tube over the carbide. Collect the gas in the test tube.

When the gas has filled the test tube, lift the tube out of the water and set fire to the gas in the test tube. *Note the luminosity of the flame.* To test the product of this combustion, close the test tube, add a little clear limewater, and shake. *What is one product of the combustion of acetylene?*

Devise and carry out a simple experiment to compare the densities of acetylene and air.

Test the liquid left in the evaporating dish with litmus papers. *What is the suspended solid?*

Write the equation for the interaction of the calcium carbide and water.

2. ***Methane*** (**CH_4**). Mix thoroughly about 4 cc. of powdered sodium acetate ($NaC_2H_3O_2$) with the same quantity of soda lime ($NaOH + Ca(OH)_2$). Place the mixture in an ignition tube. Clamp the tube near the mouth so that it is held in a horizontal position and insert a one-hole stopper fitted with an L-tube pointing upward. Tap the ignition tube to make the mixture settle and leave a space above the mixture to the end of the tube.

Heat the tube with a moving flame in order not to melt the glass. Test the gas by slipping a test tube over the vertical part of the L-tube (Fig. 82). After a minute or two remove this test tube full of gas a little distance away and see whether it burns quietly. When a sample of the gas burns quietly, bring a flame to the end of the outlet tube. *Note the structure of the flame and its luminosity.*

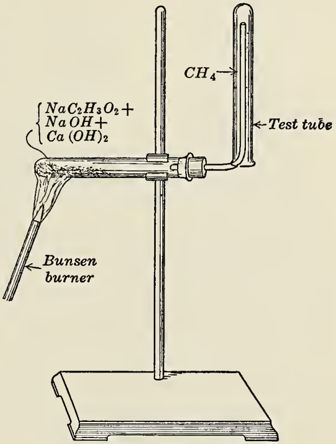

Fig. 82. Making methane.

Hold a clean dry bottle inverted over the flame for a minute and note the interior surface of the bottle. *What are the drops that appear on the inside of the bottle?*

Quickly pour about 20 cc. of limewater into the bottle, cover it, and shake it. *What is the gaseous product of burning methane?*

Write the equation for the preparation of methane (considering soda lime as sodium hydroxide), *and also the equation for the combustion of methane.*

Summarize the observed properties of acetylene and methane. Make labeled diagrams of the apparatus in your notebook.

EXPERIMENT 64

Gasoline and Kerosene

(*Optional Experiment*)

Apparatus. — Hydrometer. Baumé scale for light liquids. Hydrometer jar or cylinder. Tripod. Bunsen burner. Asbestos gauze. Beaker. Watch glass. Wide-mouth bottle with cork. Iron dish with sand. Ring stand. Thermometer. Evaporating dish.

Materials. — Gasoline (petrol). Kerosene. Wood splint.

Introduction. Gasoline and kerosene are both hydrocarbons which are prepared by the fractional distillation of crude oil. Other products which are obtained in this process are benzine, lubricating oils, vaseline, and paraffin. Sometimes coke is also prepared from crude oil. All petroleum products are mixtures of hydrocarbons; this applies, for instance, to kerosene and gasoline, which has approximately the composition C_7H_{16}. They are not chemical compounds and have no definite composition. In recent years the demand for gasoline as a fuel for internal-combustion engines has made it necessary to increase the production. So it has come about that some of the less volatile, more complex hydrocarbons in petroleum have been subjected, under high pressure, to a gasoline-refining process, called "cracking," and this has increased our supply of gasoline. The specific properties of gasoline depend upon the method of manufacture, the process of distillation, and the particular source of the crude oil.

Directions. a. ***Specific gravity.*** Although this test is not very important in determining the properties of gasoline and kerosene, yet it is worth while applying since it is an easy method of detecting differences in the commercial materials. The regular commercial hydrometer (Fig. 83) may be used to determine the specific gravity. Since the scale of the instrument is, as a rule, made for 60° F. (15.6° C.), it is usually easier to get the liquid at this temperature than to make the necessary corrections. After the specific gravity has been approximately determined, raise the hydrometer nearly down to the bulb, and wipe the stem clean; then carefully allow it to sink in the liquid. This must be done in such a way that the stem of the hydrometer is not wet above the point corresponding to the specific gravity of the liquid. Now read the mark on the stem corresponding to the level of the liquid. In this way determine the specific gravity of gasoline and kerosene

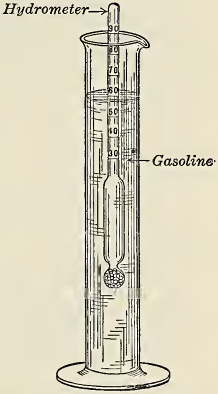

Fig. 83. Finding the density of gasoline.

Note. Commercial hydrometers are often graduated according to the Baumé Scales. The scales are divided into divisions called degrees (°); the scale for liquids lighter than water begins at 10°, which corresponds to a specific gravity of 1, and increases as the specific gravity decreases. *Degrees Baumé can be converted into specific-gravity readings by the following equation:*

$$\text{Specific Gravity} = \frac{140}{130 + \text{Degrees Baumé}}$$

Commercial gasoline (petrol) ranges from 50° Bé to 90° Bé and over.

b. *Volatility.* This is probably the most important single test of gasoline. We may roughly compare the volatility of various samples of gasoline and of kerosene by the following method: Place ten drops of gasoline on a watch glass and set the glass on a beaker half filled with boiling water.

Caution. Gasoline vapor is inflammable. Keep it away from all flames.

Note the length of time required for its complete evaporation. Repeat the experiment, using 10 drops of kerosene.

Compare the volatility of gasoline and kerosene.

Combustion of gasoline. Warm a wide-mouth bottle and then pour into it *one* drop of gasoline. Cork the bottle and shake it vigorously. Remove the cork and bring a lighted splint to the mouth of the bottle. If there is no decided result, remove the splint, add another drop of gasoline, and repeat the operation. Continue in this way to add gasoline, one drop at a time, until the vapor burns with explosive violence. *What was in the bottle besides gasoline? What is meant by an "explosive mixture"?*

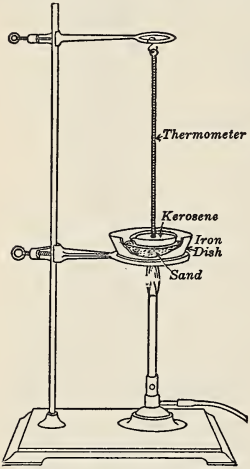

Fig. 84. Apparatus for determining the flash point of kerosene.

Flash point of kerosene. Arrange a porcelain dish on a sand bath as in figure 84. Fill the dish with kerosene to within half a centimeter of the top, and adjust the thermometer in the center of the liquid so that it almost reaches the bottom but does not quite touch the dish. Put the Bunsen burner under the center of the dish and allow the apparatus to get warm. From time to time apply a little test flame by passing it slowly across the dish about a centimeter above the liquid and just in front of the thermometer. Continue in this way until the vapor above the liquid ignites with a slight flash. The lowest temperature at which a liquid does this, as shown by a thermometer, is called the **flash point.** Repeat the experiment several times to make sure that you have found the lowest temperature at which the flash can be obtained.

Note. Most states have laws which forbid the sale for illuminating purposes of oil which has a flash point lower than about 110° F.

EXPERIMENT 65

Alcohol — Preparation and Properties

Apparatus. — A 2-liter bottle or flask. Wide-mouth bottle with 2-hole stopper. Drying tube. Boiling flask with side tube. Tripod and asbestos gauze. Ring stand and clamp. Liebig condenser. Test tubes. Bunsen burner or, better, an electric heater. Thermometer. Watch glass. Rubber tubing. Glass tubing.

Materials. — Corn sirup or molasses. Yeast cake. Limewater. Sodium hydroxide (stick or pellet form). Glass beads. Asbestos wool. Iodine. Sodium hydroxide solution. Litmus papers.

Fermentation.* Dissolve about 200 cc. of corn sirup or molasses in 2 liters of water in a large bottle or flask (Fig. 85). Crumble a yeast cake and mix it with water, making a thin paste. Then add it to the sugar solution. Connect this bottle with another containing limewater, as shown in the figure. The bulb tube contains sticks of sodium hydroxide to absorb from the air any carbon dioxide which might affect the limewater. Set the apparatus aside in a warm place (about 30° C.) for 2–3 days. The change of sugar solution (glucose) into alcohol and carbon dioxide is represented by the following equation:

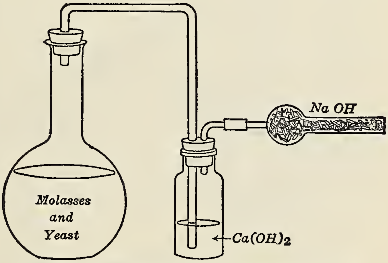

Fig. 85. Fermentation of molasses in the laboratory.

$$\underset{\text{glucose}}{C_6H_{12}O_6} \xrightarrow{\text{enzyme}} \underset{\text{alcohol}}{2C_2H_5OH} + 2CO_2\uparrow$$

Distillation. After the sugar solution has fermented, pour off the clear liquid and give each student or group of students about 150–200 cc. The apparatus for separating the alcohol from the solution may be set up as in figure 86. To prevent bumping, it is well to add a few glass beads or pieces of red-hot pumice stone to the boiling flask. In connecting the condenser to the water supply, be sure to have the cold water enter the jacket at the lower tube.

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* This part of the experiment may well be done by the instructor or a small group of students for the whole class.

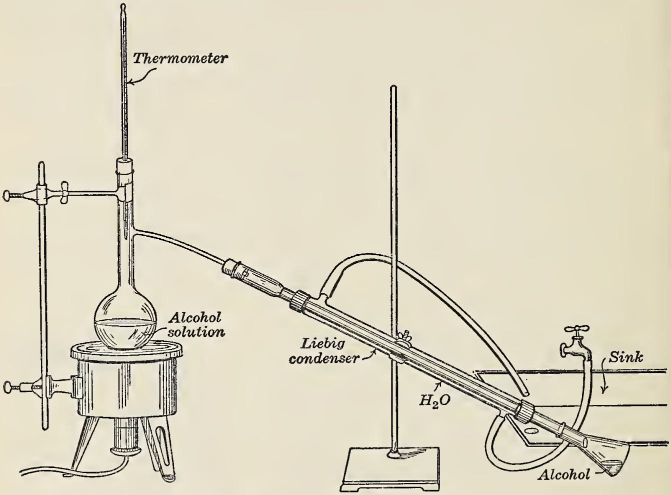

Fig. 86. Fractional distillation of alcohol.

Record the temperature at which the first few drops of liquid appear in the condenser and again when about one-half of the liquid has distilled over.

Properties. As soon as a few drops of the distillate have been collected, pour them on a watch glass. *Note the odor of the distillate.* Test it with litmus papers. Saturate a bit of asbestos wool with the liquid and try to ignite it. To recognize one product of burning alcohol, hold a cold wide-mouth bottle over the flame. Close the bottle, pour in a little limewater, and shake the contents. *Make an equation for the combustion of ethyl alcohol* (C_2H_5OH).

Test. Add 3–4 cc. of iodine solution to about 5 cc. of the distillate and then add sodium hydroxide solution until the brown color disappears. Warm the mixture and set it aside for a few minutes. The precipitate is **iodoform** (CHI_3) and shows the presence of ethyl alcohol. *Record the color and odor.*

Summarize the properties of alcohol which you have observed. Name two substances which result from the fermentation of sugar.

EXPERIMENT 66

Determination of the Acidity of Vinegar

(*Quantitative Experiment*)

Apparatus. — Burette (50-cc. grad. $\frac{1}{10}$ cc.). Pipette, volumetric (10-cc.). Ring stand and clamp.

Materials. — 100 cc. of several kinds of vinegar, such as cider and distilled. 0.1 N. solution of sodium hydroxide. Phenolphthalein solution (a few cc.).

Introduction. Vinegar is still made by allowing fruit juices to ferment to alcohol and the alcohol to ferment to acetic acid. It seems that in the presence of a certain microörganism the alcohol is oxidized by the air to acetic acid.

$$\underset{\text{alcohol}}{C_2H_5OH} + \underset{\text{(air)}}{O_2} \longrightarrow \underset{\text{acetic acid}}{CH_3COOH} + H_2O$$

The product of this process, vinegar, contains from 3 to 6 per cent of acetic acid. The Federal statutes require that all vinegar sold must contain not less than 4 grams of acetic acid per 100 cc. of vinegar. The amount of acid present may be determined by titrating a known volume of the sample of vinegar against a solution of sodium hydroxide of known concentration.

Directions. Prepare a normal solution of sodium hydroxide by dissolving 40 grams of sodium hydroxide in distilled water and diluting to 1 liter. Then take 10 cc. of this normal solution and dilute it again to 100 cc. with distilled water. This gives a dilute solution of sodium hydroxide of which 1 cc. contains 0.004 grams of sodium hydroxide.

By means of a graduated pipette, place exactly 10 cc. of the sample of vinegar in a small flask and then add about 50 cc. of distilled water. To this dilute acid solution add 2 or 3 drops of the indicator, phenolphthalein solution. Now fill the burette with the tenth-normal sodium hydroxide solution and proceed to titrate the diluted vinegar solution with the basic solution. Run this basic solution slowly, drop by drop, into the flask until the solution is neutral, following the same procedure as that used in Experiment 20. The liquids should be thoroughly mixed after each addition of the sodium hydroxide solution. Consider this as a preliminary test and now repeat the titration with 3 samples of the vinegar to be tested. The equation for this reaction is as follows:

$$\text{Na}\overset{40}{\text{O}}\text{H} + \underset{\text{acetic acid}}{\text{CH}_3\overset{70}{\text{C}}\text{OOH}} \longrightarrow \underset{\text{sodium acetate}}{\text{CH}_3\text{COONa}} + \text{H}_2\text{O}$$

Calculations. It will be convenient to record your data and results in your notebook in tabular form as follows:

Data:

Concentration of NaOH solution used		0.1 N.
Wt. of NaOH in 1 cc. of solution		0.004 g.
Vol. of NaOH sol. used to neutralize 10 cc. vinegar . . .	(1)	—cc.
	(2)	—cc.
	(3)	—cc.
Mean . .		cc.

Calculation:

Wt. of NaOH contained in mean volume used	g.
Wt. of CH_3COOH in 10 cc. of vinegar (calculated)	g.
Wt. of acetic acid in 100 cc. of vinegar	g.

EXPERIMENT 67

Soap — Its Preparation and Uses

Apparatus. — Evaporating dish. Tripod and asbestos gauze. Bunsen burner. Stirring rod. Test tubes and rack.

Materials. — Alcohol. Vegetable oil (olive, cottonseed, or cocoanut). Sodium hydroxide solution (30%). Calcium chloride solution. Kerosene.

Introduction. Sodium hydroxide (NaOH) is one of the strong bases (an alkali). When fats or vegetable oils are boiled with it, they are saponified, *i.e.* converted into soap. These fats are chiefly glyceryl esters of organic acids. On boiling, the fat is decomposed by the sodium hydroxide into glycerol ($C_3H_5(OH)_3$) and the sodium salt of the organic acid, which is soap. The following equation represents such a reaction:

$$\underset{\text{glyceryl stearate}}{\overset{\text{Fat}}{(C_{17}H_{35}COO)_3C_3H_5}} + \overset{\text{Alkali}}{3\ NaOH} \longrightarrow \underset{\text{sodium stearate}}{\overset{\text{Soap}}{3\ C_{17}H_{35}COONa}} + \overset{\text{Glycerol}}{C_3H_5(OH)_3}$$

Directions. a. *Preparation.* Add 10 cc. of alcohol to about 2 teaspoonfuls of oil (olive, cottonseed, or cocoanut) in an evaporating dish. Heat the dish with a small flame which barely touches the dish. Then add 5 cc. of a 20 per cent solution of sodium hydroxide and continue heating and stirring the contents constantly until the odor of alcohol is no longer perceptible (Fig. 87). The pasty mass which remains in the dish is the soap mixed with glycerol.

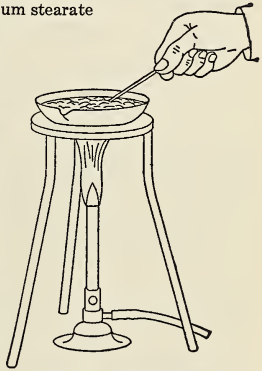

Fig. 87. Making soap.

NOTE. To save time in this experiment, alcohol is used as a common solvent for the oil and the alkali. In commercial work it is not used.

b. *Uses.* Wash your hands with a little of the soap which you have just made. *Does it lather readily?* When too much alkali has been used, the soap makes the hands rough. An excess of oil makes the soap greasy. *What about the quality of your soap?*

Make a half test-tubeful of soap solution, add to it half as much calcium chloride ($CaCl_2$) solution, and shake the liquid. An insoluble calcium soap is formed. This insoluble soap, which is formed whenever soap is used with "hard" water, is useless for cleaning purposes.

Put 1 cc. of kerosene into 10 cc. of water in a test tube and shake the mixture. Note that the oil is broken up into tiny globules, which form an **emulsion.** Let the test tube stand for a minute to see whether the emulsion remains; then add about 3 cc. of soap solution to the mixture and shake again. *What effect does the soap have on the permanency of the emulsion?* In this same way soap forms an emulsion with the oil. It is this oil that makes dirt cling to the fibers of clothing. This action loosens the dirt so that it can be easily removed by mechanical washers.

EXPERIMENT 68

Removal of Spots and Stains

Apparatus. — Beakers. Test tubes and rack. Bunsen burner.

Materials. — Samples of various kinds of cloth (cotton, wool, silk, and rayon) spotted with grease, sugar, ink, iron rust, etc. Carbon tetrachloride, turpentine, oxalic acid, Javelle water, etc.

Introduction. The simplicity of the cleansing process varies with the character of the stain. For example, the removal of a grease spot from cloth is merely a process of solution and absorption; but the removal of a fruit stain or of an ink spot from cloth often involves a careful study of the cloth itself as well as of the various bleaching reagents.

In general, there are three methods of treatment: (1) **warm water** will remove sugar, glue, blood, if not too old; (2) **gasoline, carbon tetrachloride,** and **turpentine** will act as solvents for grease and paint and so can be used to remove such spots;

(3) bleaching agents, such as oxalic acid or citric acid, followed by bleaching powder may be used to remove ink spots. Of course, it is quite important to remember that gasoline vapor is very inflammable and should never be used in the house. Some absorbent material must be placed beneath the goods to be cleaned in order that the grease may be washed through by the gasoline. Otherwise, rubbing a little gasoline on a spot merely spreads the grease over a larger area. In using bleaching agents it is necessary to remove with dilute ammonia water the traces of such agents, which might rot the fibers of the cloth.

It is always well to test the treatment on a small sample of the goods before trying to remove the spot or stain from a garment. In general, greater care must be exercised with woolen and silk goods than with cotton, and especially with colored goods. This is due to the fact that protein material (silk and wool) is attacked by many chemicals.

The following table will be useful for reference:

Stain	Removed by
Sugar, glue, blood	Warm water
Grease	Carbon tetrachloride or gasoline; followed by warm soapsuds and ammonia water
Paint, varnish	Turpentine (except on silk)
Tar, pitch	Soap and oil; followed by turpentine
Inks	Warm oxalic or tartaric acid (20%) or salt and lemon
Iron rust	Warm oxalic or citric acid (10%)
Fruit, tea, and coffee	Hot water. Javelle water *
Acids	Ammonia water to neutralize the acid; then wash with water
Grass	Sponge with alcohol or ammonia water

Directions. Take samples of 3 or 4 different kinds of cloth and stain them with ink, acid, sirup, and oil. Treat each sample according to the methods just described. Cut the samples down to some convenient but uniform size and mount with a label in your notebook. Show a sample of the piece of cloth before and after treatment. Also describe briefly just what the treatment was in each case.

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* Javelle water is made by dissolving a pound of washing soda ($Na_2CO_3 \cdot 10 H_2O$) in a quart of boiling water. Add a quarter of a pound of chloride of lime ($CaOCl_2$) to a quart of hot water. Mix the two solutions and decant the clear solution of sodium hypochlorite ($NaOCl$) for use.

EXPERIMENT 69

Textile Fibers

(*Class Experiment*)

Apparatus. — Compound microscope. Test tubes and rack. Bunsen burner.

Materials. — Samples of cotton, wool, silk, linen, and rayon cloth. Samples of cotton and wool mixed goods, cotton and linen mixed goods. Sodium hydroxide solution (20%). Alcoholic solution of fuchsin. Dilute ammonium hydroxide. Nickel hydroxide solution. Dilute hydrochloric acid.

Introduction. Clothing stands next to food as a necessity for the human race. Textiles are, in general, made up of fibers which are either plant products, such as cotton and linen, or animal products, such as wool and silk. In recent years many processes have been developed by which the fibers of cotton, which is cheap, may be made to look very much like the more expensive silk fibers. However, these fibers can always be identified by chemical or microscopic examination. In this experiment we shall learn some of the chemical tests which can be applied in distinguishing these textile fibers.

Directions. **a.** *Microscopic study of fibers.** Study carefully the appearance of fibers of cotton, wool, silk, linen, and rayon under the microscope. A projection microscope will greatly aid in this experiment. (Compare with figure 272 on page 461 in B. & C., NEW PRACTICAL CHEMISTRY.)

b. *Cotton and wool.* Place in separate test tubes a sample (3 cm. × 3 cm.) of cotton cloth, woolen cloth, and mixed goods.

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* The instructor will have the slides prepared and set up. These slides can be purchased already prepared.

Cover each with sodium hydroxide solution (20%) and boil for 2–3 minutes. Add water when necessary to keep the cloth covered with liquid.

Caution. **Take great care not to let the hot alkali spatter over on your hands.**

Which one of these fibers is completely soluble in sodium hydroxide solution?

c. ***Cotton and linen.*** Wet a sample of cloth (part linen) with a 1 per cent alcoholic solution of fuchsin, wash well in water, and then moisten with ammonium hydroxide. The linen (flax) fibers in the cloth will be colored red, while the cotton remains uncolored.

d. ***Wool and silk.*** Put strips of wool and silk in separate test tubes and cover each with a solution of nickel hydroxide.* After 2 minutes remove the residue and boil for another 2 minutes in a 1% solution of hydrochloric acid. *Which of the samples remains undissolved?*

e. ***Silk and rayon.*** Since rayon is an artificial silk manufactured in various ways from cellulose, it is easy to distinguish it from real silk. Cellulose is the vegetable material found in the woody fiber of plants, and real silk is an animal fiber which is made of complex nitrogen compounds. Apply the burning test as follows: Ravel out threads from samples of real silk and rayon and separate the distinct kinds of threads. Burn each of the distinct kinds of threads in small test tubes and note the odor. *What is the distinctive odor produced by burning real silk?*

f. ***Silk, wool, and cotton.*** *How would you detect the presence of silk, wool, or cotton in a sample?*

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* Nickel hydroxide solution may be prepared by dissolving 5 g. of nickel sulfate in 100 cc. of water and then adding a solution of sodium hydroxide until the nickel hydroxide is completely precipitated. Wash the precipitate well and dissolve it in 25 cc. of concentrated ammonium hydroxide. Then add 25 cc. of distilled water.

EXPERIMENT 70

Components of Foods

Apparatus. — Test tubes and rack. Bunsen burner. Mortar and pestle. Large beaker. Funnel. Crystallizing dish. Iron spoon.

Materials. — Starch. Iodine solution. Glucose. Fehling's solution (two solutions). Concentrated nitric acid. Concentrated ammonium hydroxide. Egg. Cornmeal. Peanuts. Oatmeal. Ether. Filter papers.

Introduction. It is customary to divide the food we eat into four classes: (1) **carbohydrates,** such as starch and sugar, which furnish both heat and energy and help in building fats in the body; (2) **fats,** such as butter, meat fat, and olive oil, which produce both heat and energy; (3) **proteins,** such as lean meat and the whites of eggs, which are chemically very complex substances rich in nitrogen and which replace the worn-out muscle tissue. Finally, there are various (4) **mineral compounds,** which serve the body in a great variety of ways but are needed in extremely small quantities. In addition to these substances it has been shown that very minute amounts of certain other compounds seem to be essential for the growth and healthy condition of animals. These substances are called **vitamins.** In this experiment we shall learn the tests for the four components of our food first mentioned.

Directions. **a.** *Starch.* Put a pinch of starch into a test tube and fill the tube half full of water. Shake the contents and boil. *Has the starch changed?* Cool the liquid in running water and, as soon as it is cold, divide it into two parts. To one add a drop of iodine solution (iodine in potassium iodide). The blue color which is produced is the iodine **test for starch.**

b. *Glucose.* Dissolve about 1 cc. of glucose in 10 cc. of water. Add 5 cc. of an alkaline solution of copper sulfate (Fehling's solution) and boil for a few minutes. The red precipitate (cuprous oxide, Cu_2O) is the characteristic **test for glucose.**

c. *Protein.* Place a few bits of the white of a hard-boiled egg in a test tube and add a few drops of concentrated nitric acid. Wash off the acid with water and pour off the liquid. Then pour a few drops of ammonium hydroxide on the egg. The yellow color is a **test for proteins** (nitrogenous compounds).

d. ***Fats and oils.*** Half fill a test tube with cornmeal or crushed peanuts and pour in enough ether or benzine (away from flames) to cover the solid to a depth of a centimeter. Stand the test tube in a beaker of hot water and occasionally shake the contents. Filter and let the filtrate evaporate spontaneously in a crystallizing dish. The fat or oil extracted remains in the dish.

e. ***Mineral matter.*** Place about half a teaspoonful of oatmeal on a porcelain crucible cover and heat it (under the hood) until no more fumes are given off. Then heat it strongly to burn off the carbon. The white ash contains various mineral compounds.

f. ***Examination of a food.*** Apply these tests to one or more foods, such as lean meat, bread, beans (dried), and cheese. *Record your results in tabular form.*

EXPERIMENT 71

Food Components of Milk

(*Optional Experiment*)

Apparatus. — Evaporating dish. Short stirring rod. Steam bath. Tripod. Bunsen burner. Ring stand. Horn-pan balance and weights. Test tubes and rack. Funnel.

Materials. — Sample of milk (from home). Acetic acid. Junket tablets. Fehling's solution. Concentrated nitric acid. Concentrated ammonium hydroxide.

Introduction. Milk is one of the most valuable of foods. The average of a large number of analyses shows that its approximate composition is as follows:

Water	87 %	Fat	4%
Protein	3.3%	Lactose	5%
Mineral matter	0.7%		

From this it will be seen that milk contains proteins, fats, and carbohydrates, which are essential in any well-balanced diet. For persons of all ages milk forms one of the most economical sources of proteins, fats, mineral compounds, and vitamins. For growing children there is nothing which so adequately and economically supplies all of the essential foods.

Milk is a natural emulsion of tiny globules of fat. On standing, the fat (cream) comes to the top. The difference between whole milk and skim milk is readily seen when a sample is examined under the microscope (Fig. 88).

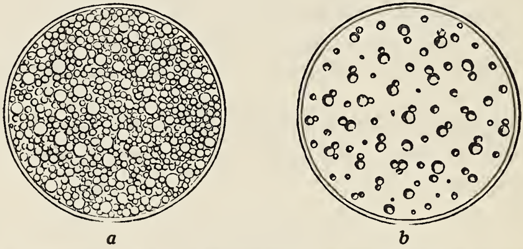

Fig. 88. Whole milk (*a*) and skim milk (*b*) as seen under the microscope.

Directions. a. *Percentage of solids and water.* To determine the percentage of water in milk, we have only to evaporate to dryness a known weight of milk. During this process a scum forms over the surface of the liquid, which retards evaporation, and so it is well to use a short stirring rod occasionally to break up the scum.

Weigh a small, clean evaporating dish together with a short stirring rod and then pour into it about 20 cc. of milk and weigh again. Evaporate over a steam bath to dryness. As soon as the residue is perfectly dry, wipe dry the outside of the dish. Then cool and reweigh. From these results compute the percentages of solids and water in the sample of milk taken. (Cow's milk usually contains not less than 12 per cent of solids.)

b. *Protein.* The principal protein in milk is casein, which is used in the manufacture of cheese. Add 2–3 drops of acetic acid (vinegar may be used) to 10 cc. of milk. When thoroughly mixed and allowed to stand, the casein separates in the form of a curd. When milk sours, the lactic acid formed in the milk causes a natural curdling. Prove that the curd is a protein (Exp. 70).

Another method of separating the casein is by means of rennet. "Junket" tablets contain dried rennet. Dissolve about one-half of such a tablet in water and add the solution to about 100 cc. of warm milk. Again the casein will separate as a curd.

There is another protein in milk which remains in solution when the casein is separated either by an acid or by rennet. This is **albumin,** which is much like the white of egg. Filter off the milk curd (casein) and boil the clear liquid (whey). This coagulates the albumin in the form of white flakes. Prove that the tough scum which forms on the surface of boiled milk is coagulated albumin (a protein).

c. *Sugar of milk* (*lactose*). The filtrate left after the casein and albumin have been coagulated and removed contains the sugar of milk. By evaporation of this filtrate we obtain a residue. The taste shows that this sugar is only about one-sixth as sweet as cane sugar. Test it with Fehling's solution.

d. *Butter fat.* The most valuable component of milk is doubtless the **butter fat.*** The most practical method of determining the percentage of butter fat is the **Babcock test.** In this process a definite quantity (17.6 cc.) of milk is treated with sulfuric acid (17.5 cc., sp. gr. 1.83), which dissolves the casein of the milk and thus facilitates the separation of the fat. This mixture is then whirled in a centrifugal machine, and the fat collects on the surface. The necessary apparatus is shown in figure 89. The test bottle is so made that the graduations on the neck indicate the percentage of fat directly. Further directions are furnished with the apparatus.

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* Since cream will rise on a sample of milk in 5 minutes sufficiently to destroy the uniformity of the sample, great care must be used to get a fair sample. Mix the cream with the milk thoroughly by pouring the milk from one vessel to another several times.

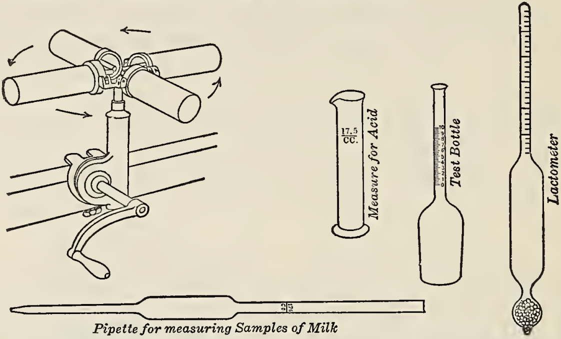

Fig. 89. Milk-testing outfit. Centrifuge. Babcock test bottles and lactometer.

EXPERIMENT 72

Extracting Metals

Apparatus. — Ignition tube (Pyrex), $6'' \times \frac{3}{4}''$. 1-hole stopper with bent delivery tube. Test tubes. Ring stand with ring and clamp. Bunsen burner (or Fisher form). Pipe-stem triangle. Porcelain crucible. Porcelain battery top. Tumbler. 2 dry cells or 110-volt d-c. line with 60-watt lamp in series.

Materials. — Metallic oxide, such as lead oxide, ferric oxide, or zinc oxide. Metallic sulfide (galena). Charcoal (powdered). Limewater. Saturated copper sulfate solution. Two electric-light carbons. Dilute nitric acid (1:1).

Introduction. There are three general methods of extracting metals from the ores. *First,* by roasting the ore with a flux that will unite with the rocky material in which the ore is found. *Second,* by reducing the ore by heating it with some form of carbon. In this process the carbon unites with the oxygen of the ore and goes off as carbon monoxide gas. In case it is a carbonate or a sulfide, it must first be heated to obtain the oxide, and then the oxide is reduced with carbon. *Third*, by electrolysis: the ore is fused and then the metal is deposited by the electric current at the cathode.

Iron is obtained by reduction in a blast furnace with coke, limestone being used as a flux. Copper, lead, and zinc are obtained by roasting and reduction. Aluminum and magnesium are obtained by electrolysis. Copper is purified by electrolysis.

In this experiment we shall first reduce litharge (PbO) with carbon; second, we shall roast and reduce lead sulfide (PbS); and third, we shall deposit pure copper from a solution of copper sulfate ($CuSO_4$).

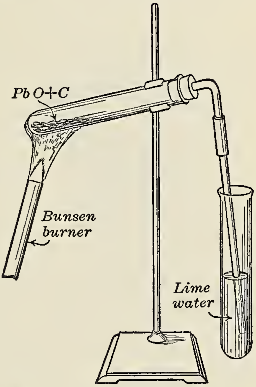

Fig. 90. Reducing lead oxide with charcoal.

Directions. a. ***Reduction.*** On a sheet of paper mix 15 grams of lead oxide with about 1 gram of powdered charcoal. Slide this mixture into an ignition test tube and insert a stopper and delivery tube as shown in figure 90. Fill another test tube about $\frac{1}{3}$ full of limewater. Let the delivery tube just dip below the surface of the limewater.

Heat the ignition test tube gradually by waving the Bunsen flame about the tube, beginning at the upper part of the mixture. Finally heat the mixture as hot as possible with the Bunsen flame. The Fisher form of the Méker burner is excellent for this experiment.

What change do you notice at first in the limewater? What gas is produced in this process?

Keep on heating until this gas ceases to be given off. *How do you test this point?* Then remove the test tube containing the limewater and allow the ignition tube to cool. Remove the stopper and delivery tube and empty very carefully the contents of the ignition tube upon the base of the ring stand. *Write the equation for this reduction process.*

b. *Roasting and reduction.* Put a little lead sulfide (PbS) in a porcelain crucible and mount on the pipe-stem triangle as shown in figure 23. Heat the crucible as hot as possible for a few minutes. Cautiously smell the gas which is produced. When an ore is roasted in air, it reacts with the oxygen. The residue left is the oxide. *What is the gas evolved? Write the equation for this roasting process.*

If time permits, reduce with charcoal, as in part (a), the residue left.

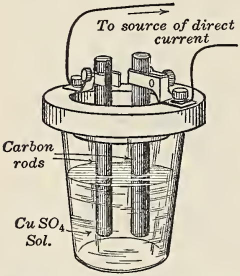

Fig. 91. Electrolysis of copper sulfate solution.

c. *Electrolysis.* Place a saturated solution of copper sulfate ($CuSO_4$) in a glass tumbler and insert two carbon rods in the clamps of the battery top, as shown in figure 91. Connect the terminals with a source of direct current such as 2 dry cells or a 110-volt d-c. line with a 60-watt lamp in series. Allow the current to pass through the copper sulfate solution for about 5 minutes. Then disconnect the electric current and lift the carbon electrodes out of the solution. Rinse and examine each carefully. *What substance was deposited on one of the electrodes?* Was it on *the anode or the cathode?* Place the electrode with the deposit in a test tube containing a little dilute nitric acid. *What does this show the deposit to be?*

Questions on the theory. *Write the equation for the dissociation of copper sulfate. How do the copper ions become copper atoms? How do you express these facts in terms of the electron theory?*

EXPERIMENT 73

Aluminum Hydroxide — Preparation and Uses

(Optional Experiment)

Apparatus. — Test tubes and rack. Wide-mouth bottle or glass cylinder.
Materials. — Aluminum sulfate. Limewater. Clay. Cochineal or indigo. Dilute ammonium hydroxide.

Preparation. Prepare about 5 cc. of aluminum sulfate solution ($Al_2(SO_4)_3$) or the same quantity of any aluminum salt and add limewater until a precipitate is formed. *Describe the appearance of the precipitate* ($Al(OH)_3$) *and write the equation.*

Clarifying muddy water. Fill a glass bottle or cylinder about two-thirds full of water and render the water turbid with a little finely pulverized clay. Pour in about 10 cc. of aluminum sulfate solution and mix it thoroughly with the turbid water. Then add slowly, without stirring, 20 cc. of limewater. Set the cylinder aside to settle. *What precipitate was formed in the turbid water? Explain how the suspended particles of clay are removed.*

Mordant. Take about 5 cc. of water in a test tube and add enough cochineal or indigo solution to color the water faintly. Add an equal volume of aluminum sulfate solution and enough ammonium hydroxide to precipitate the aluminum hydroxide. Shake the test tube and allow the contents to settle. *Note the effect on the color of the solution.*

NOTE. The particles of dyestuff are much smaller than those of the clay, but the aluminum hydroxide collects them. In dyeing cloth, aluminum hydroxide is sometimes used as a binder (mordant) between the cloth and the dye.

EXPERIMENT 74

Cobalt Nitrate Tests for Metals

Apparatus. — Forceps. Blowpipe. Bunsen burner.

Materials. — Charcoal block (or block of plaster of Paris). Zinc sulfate. Solution of cobalt nitrate (5%). Aluminum sulfate. Magnesium carbonate. Unknowns.

Introduction. Cobalt nitrate ($Co(NO_3)_2$) is often used in analytical work to test for certain metals because it unites with metal oxides, forming chemically colored compounds. Thus, aluminum compounds, when converted into the oxides by heat before the blowpipe flame, give a blue coloration when further heated with cobalt nitrate. When this is done, the oxide forms cobalt aluminate. In a similar way zinc compounds, when heated with cobalt nitrate, form double oxides of cobalt and zinc, which have a similar color.

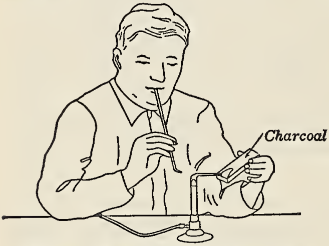

Fig. 92. Using a blowpipe flame on charcoal.

The use of the blowpipe. It will be well to practice using the blowpipe before beginning the experiment. Use a Bunsen burner with its air inlets closed to give a luminous flame. Then turn the gas down so that the flame is only 4 or 5 cm. high. Put the tip of the blowpipe in the middle of the flame and blow in such a way that a small blue flame is produced. It should now be possible to turn this little Bunsen flame downward on the object to be heated. After a little practice it will be found possible *to do these things at the same time:* 1. to keep a steady stream of air in the blowpipe; 2. to breathe through the nose; 3. to keep a surplus of air in the cheeks.

Zinc. Make a cavity in a charcoal block with the top of the forceps. Place a little zinc sulfate in this cavity and heat it as hot as possible at the tip of a small blowpipe flame (Fig. 92). *After the residue has cooled, record its color.*

Put a drop or two of cobalt nitrate solution on the residue and reheat it intensely. *After it has cooled, record the color of the mass that remains on the charcoal block.*

Aluminum. Make a fresh cavity in the block and place in it some aluminum compound, as aluminum sulfate, and repeat the test made with the zinc compound. *Record the characteristic coloration which is obtained.*

Magnesium. Repeat (a), using some compound of magnesium, such as magnesium carbonate or sulfate. Heat the mass until it glows brightly. Cool and moisten the fused mass with only a very little cobalt nitrate solution. Reheat intensely and after the mass has cooled, note carefully the delicate coloration.

Lead. A convenient test for lead is the charcoal test. Heat a fragment of lead in the oxidizing flame and note the result. Mix lead oxide with soda, and heat on charcoal in the reducing flame (Fig. 93). Note the metallic globule.

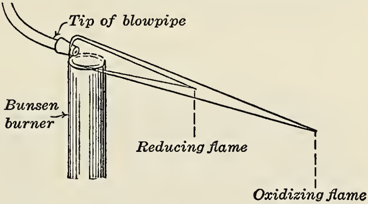

Fig. 93. Oxidizing and reducing flames.

Unknown. Obtain from the instructor some compound of aluminum, zinc, or magnesium and identify the metal with the blowpipe and cobalt nitrate solution. *Record in your notebook, preferably in tabular form, your results as follows:*

TABLE

Compound Used	Color of Residue after First Heating	Color of Residue after Heating with $Co(NO_3)_2$
Unknown . . .		

EXPERIMENT 75

Relative Activity of Metals

Apparatus. — Test tubes and rack.

Materials. — Five strips of zinc (10 × 1 cm.). Sandpaper. Solutions of copper sulfate, lead nitrate, silver nitrate, zinc nitrate, and mercurous nitrate. Dilute sulfuric acid. 3 strips of lead. 4 strips of copper.

Zinc. Prepare 5 strips of zinc (10 × 1 cm.) and clean each strip with sandpaper. In separate test tubes place about 5 cc. each of solutions of lead nitrate, copper sulfate, silver nitrate, mercurous nitrate, and dilute sulfuric acid. Label each tube with the formula of the substance it contains and also with the symbol of the positive ion in each case. Place a strip of zinc in each solution in such a way that the top of the strip bends over the edge of the tube (Fig. 94).

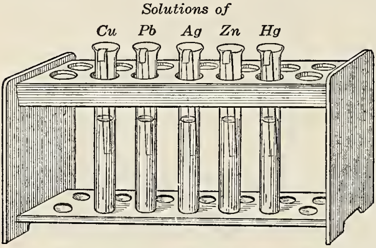

Fig. 94. Strips of metal partly immersed in various solutions.

After 5–10 minutes withdraw the strips and examine them minutely. Remember that finely divided metals present a very different appearance from the same metals in compact polished condition. Tabulate your results as follows:

Metal Strip	Solution	Deposit
Zinc . . . . etc.	Lead nitrate	Lead

Lead. Prepare 3 strips of bright, clean sheet lead. Place in separate test tubes about 5 cc. each of solutions of mercurous nitrate, silver nitrate, and copper sulfate. Label each tube as in (a) and place a strip of lead in each solution. *Examine later and tabulate the results.*

Copper. In the same way prepare 4 strips of copper and place them in another set of test tubes containing solutions of lead nitrate, silver nitrate, zinc nitrate, and mercurous nitrate. *Note any change in the color of the solution left in the test tube. Record negative as well as positive results. For each case giving a positive result write the equation showing the ions. In those cases which give a negative result, explain why it is so.* Compare your results with the REPLACEMENT SERIES OF METALS on p. 485 in B. & C., NEW PRACTICAL CHEMISTRY.

EXPERIMENT 76

Ferrous Compounds

Apparatus. — Test tubes and rack. Bunsen burner. Funnel. Small flask and cork. Tripod. Asbestos gauze.

Materials. — Small iron nails (or steel wool). Dilute hydrochloric acid. Filter papers. Dilute ammonium hydroxide. Potassium ferrocyanide solution. Potassium ferricyanide solution. Potassium sulphocyanate solution.

Introduction. Iron forms two sets of compounds — **ferrous** compounds, in which the iron has a valence of two, and **ferric** compounds, in which the valence of iron is three. The process of changing the ferrous to ferric is called **oxidation,** which means an increase in valence. On the other hand, the process of changing from ferric to ferrous is called **reduction,** and this involves a decrease in the valence of the iron. In this experiment we shall learn how to prepare ferrous compounds and how to test for the same. In Experiment 77 we shall learn how to convert ferrous to ferric compounds and how to make the appropriate tests.

Directions. 1. ***Making ferrous chloride.*** Place in a test tube about a spoonful of small iron nails or a small bundle of steel wool and add just enough dilute hydrochloric acid to dissolve the iron. Warm the acid to keep the action going briskly. *Identify the gas given off. Account for its odor and for the insoluble black residue.*

Filter the liquid into a small flask containing about 50 cc. of boiling water. Add a few nails and cork the flask loosely, removing the cork only when pouring out some of the solution. *Note the color of the solution. Write the equation for making ferrous chloride* ($FeCl_2$).

2. ***Ferrous hydroxide.*** Pour about 5 cc. of this ferrous chloride solution into a test tube and add dilute ammonium hydroxide until the solution after shaking gives the odor of ammonia. *What is the precipitate? Write the equation for this reaction.*

Filter and open out the paper with the precipitate. Lay it aside to dry. *Note the changes in the color of the precipitate when exposed to the air.*

3. ***Tests.*** a. To about 5 cc. of ferrous chloride solution add about 1 cc. of potassium ferrocyanide ($K_4Fe(CN)_6$) solution. Note that the white precipitate immediately begins to turn blue. *Write the equation, assuming that potassium ferrocyanide ionizes as follows:*

$$K_4Fe(CN)_6 \longrightarrow 4\ K^+ + Fe(CN)_6^{----}$$

b. To another portion of ferrous chloride solution add potassium ferricyanide ($K_3Fe(CN)_6$) solution. *Note the color produced. Note whether there is a precipitate or simply a colored solution.* To determine the color of a very deeply colored solution, dilute with much water.

c. To still another portion of ferrous chloride solution add potassium sulphocyanate (KSCN) solution. The result should be negative.

Give one distinctive test for ferrous salts (Fe^{++}).

NOTE. Keep the rest of the ferrous chloride solution for Exp. 77.

EXPERIMENT 77

Ferric Compounds

Apparatus. — Small flask. Tripod. Asbestos gauze. Bunsen burner. 1-hole rubber stopper with Bunsen valve (Fig. 95). Test tubes and rack.

Materials. — Ferrous chloride solution (from Exp. 76). Concentrated hydrochloric and nitric acids. Concentrated ammonium hydroxide. Solutions of potassium ferrocyanide, potassium ferricyanide, and potassium sulphocyanate. Steel wool. Iron turnings.

Ferrous to ferric. Boil the remainder of the ferrous chloride solution (Exp. 76) with a little concentrated hydrochloric acid and nitric acid for about 2 minutes. *Note the change in color* — this is characteristic of ferric solutions. The nitric acid serves as an oxidizing agent. *Complete and balance in your notebook the equation:*

$$\text{--}\ FeCl_2 + \text{--}\ HCl + [O] \longrightarrow \text{--}\ FeCl_3 + \text{----}$$

Ferric hydroxide. To a portion of the ferric chloride solution, add an excess of ammonium hydroxide. If the precipitate is green, the above action (called oxidation) was incomplete and should be repeated. *Write the equation for the formation of ferric hydroxide* ($Fe(OH)_3$). *Explain the change in color in the ferrous hydroxide prepared in Exp.* 76.

Tests. Repeat all the tests of Exp. 76 (3), using ferric chloride solution instead of ferrous chloride. *Give two distinctive tests for a ferric salt.*

Reduction of a ferric compound. Boil in a small flask * (Fig. 95) the remainder of the ferric chloride solution with a

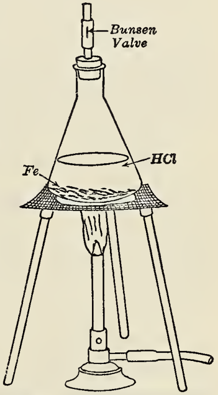

Fig. 95. Flask with Bunsen valve.

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* It is well in preparing ferrous compounds to provide the flask with a Bunsen valve. This is a short rubber tube with a slit cut in the side and plugged at the end

few drops of concentrated hydrochloric acid and some iron turnings or small iron nails. Keep the action going vigorously until the characteristic color of ferric compounds has disappeared. Pour off some of the clear solution and test quickly for the ferrous ion. *Complete in your notebook the equation:*

$$FeCl_3 + Fe \longrightarrow \_\_\_\_$$

Summarize the results of the following reagents with ferrous and ferric compounds in tabular form:

Reagents	Ferrous	Ferric
Ammonium hydroxide . .		
Potassium ferrocyanide . .		
Potassium ferricyanide . .		
Potassium sulphocyanate .		

EXPERIMENT 78

Blueprints and Inks

Apparatus. — Two photographic trays or shallow pans, enameled (3″ × 10″). Test tubes and rack. Graduated cylinder, 100 cc.

Materials. — Ferric chloride solution (4%). Potassium ferricyanide solution (10%). Oxalic acid solution (5%). Good bond paper (3″ × 4″). Absorbent cotton. Nutgall powder.

Introduction. Blueprints are commonly used in place of photographs in making copies of engineering drawings. The paper is prepared by first soaking it in a solution of potassium ferricyanide and a ferric salt and then drying it in a dark place. When a drawing on tracing cloth is placed upon such a sensitized paper and the two are exposed to the sunlight, the paper turns a brownish color, except where it is covered by the black lines. The paper is then thoroughly washed with water to remove the soluble salts. The portions that were exposed to the light turn blue, leaving the rest white. The light has reduced the ferric salt (usually ferric ammonium citrate) to the ferrous salt. In the process of washing, this ferrous salt reacts with the potassium ferricyanide to form the insoluble deep blue known as Turnbull's blue.

Directions. ***Blueprint paper.*** (a) In a dark-room or cupboard, mix in a test tube about 10 cc. of ferric chloride solution with an equal volume of oxalic acid ($H_2C_2O_4$) solution. Divide this mixture into two equal parts. Keep one portion in the dark. Expose the other to sunlight for several minutes. Then add to each portion about 4 or 5 drops of potassium ferricyanide solution.

Compare the results. In which case has a ferrous salt been produced? This is an example of photochemical action, that is, a reaction produced by light.

Complete in your notebook the following equation:

$$\_\_\ FeCl_3 + H_2C_2O_4 \longrightarrow \_\_ + \_\_\ HCl + \_\_\ CO_2 \uparrow$$

(b) Prepare in a darkened room another portion of the mixture prepared in (a) and coat a piece of nonporous bond paper with this solution. You may use a wad of absorbent cotton, soaked in the solution, to coat the paper with a thin layer of the solution. Put the paper aside in a dark place to dry. In the meantime cut a stencil from a piece of cardboard. Lay this stencil over the sensitized paper and expose both to the sunlight for a few (5) minutes.

To develop the print, float the paper on a solution of potassium ferricyanide. Then wash the paper in water until all the yellow color has been removed from the unexposed portions of the paper. Dry the blueprint and mount it in your notebook.

Complete in your notebook the following equation:

$$\_\_\ FeCl_2 + \_\_\ K_3Fe(CN)_6 \longrightarrow \_\_\_\_ + \_\_\_\_$$

In making commercial blueprint paper, ferric ammonium citrate is used because it contains both the iron salt and a reducing agent.

Making ink. Mix in a test tube a concentrated solution of ferrous sulfate with a little of a solution of powdered nutgalls (containing gallic acid). *What is the color of the mixture? What change does the mixture undergo on standing for a few minutes? When used in a pen, how does it work as ink?*

To a small portion of the ink, add some oxalic acid solution. *What is the result? What does this experiment suggest about a method of removing ink stains?*

NOTE. Circular No. 95 published by the U. S. Bureau of Standards gives much valuable information about inks.

EXPERIMENT 79

The Chlorides of Tin

(*Optional Experiment*)

Apparatus. — Test tubes. Bunsen burner.

Materials. — Tin. Concentrated hydrochloric acid. Concentrated nitric acid. Solutions of mercuric chloride and ferric chloride.

Introduction. From the position of tin in the Replacement Series of Metals [p. **485**] we should expect this metal to react with hydrochloric acid and set free hydrogen. But tin forms two chlorides, **stannous chloride** ($SnCl_2$) and **stannic chloride** ($SnCl_4$). The stannous chloride is easily oxidized to the stannic chloride, and with suitable reagents the stannic chloride can be reduced to stannous chloride. Conversely, the reagents used in these changes are themselves reduced or oxidized. Compare these compounds with the ferrous and ferric compounds in Exp. 76 and 77. Tin compounds are very important in the textile industry.

Directions. **a.** Pour upon a small piece of metallic tin (about 1 g.) in a test tube 5 cc. of concentrated hydrochloric acid and 5 cc. of water. Warm the solution if necessary to keep up a vigorous action until the tin has disappeared. *What was the gas evolved? Is the action slow or fast?*

b. To determine which chloride of tin has been formed, add a few drops of this tin solution to 5 cc. of mercuric chloride

($HgCl_2$) solution. The white precipitate formed is mercurous chloride ($HgCl$). Now add a few drops more of the tin solution and heat the mixture gently. The dark-gray precipitate is metallic mercury. *What does this prove as to the tin compound formed by the action of the hydrochloric acid on tin? Write an equation for the reaction.*

c. Dilute 1 cc. of ferric chloride ($FeCl_3$) solution with 3 cc. of water and add drop by drop some of your original tin solution until a change in color takes place. *What was the change in color?* Recall that in dilute solutions ferric salts are reddish-brown and ferrous salts are colorless. *Write the equation for the reaction. What kind of change has taken place in the tin compound?*

d. Add 10 drops of concentrated nitric acid to 5 cc. of the original tin solution and heat to boiling. Cool the solution and test it with mercuric chloride solution as in (b). *What has happened to the stannous chloride solution? Will this change be brought about by the air? How would you prevent a solution of stannous chloride from changing?*

e. Boil for several minutes a small portion of the stannic solution ($SnCl_4$) from (d) with a little granulated tin. Now add mercuric chloride solution and compare with the results in (b). *Write the chemical equation for this reaction. What kind of chemical change was this?*

EXPERIMENT 80

Salts of Lead

Apparatus. — Test tubes. Bunsen burner.

Materials. — Solutions of lead nitrate, sodium iodide, and hydrogen sulfide. Dilute nitric acid and hydrochloric acid.

Introduction. Since many of the salts of lead are insoluble, since they can easily be prepared by precipitation from solution, and since they have a distinctive color, it will be useful in identifying compounds of lead to become familiar with their appearance.

Directions. a. Dilute 3 cc. of lead nitrate solution in a test tube with 5 cc. of water and then add a solution of hydrogen sulfide. *Name and describe the precipitate. Write the equation for its formation.* Add 5 cc. of dilute nitric acid. *Does the precipitate dissolve in nitric acid?*

b. Dilute 3 cc. of lead nitrate solution with 5 cc. of water and add 10 drops of dilute hydrochloric acid. *Name and describe the precipitate. Write the equation for its formation.* Heat the solution. *What change occurs?* Cool it again under running water. *What appears? What do you conclude about the solubility of lead chloride? What other insoluble chloride have you prepared? How could you distinguish between these chlorides?*

c. To 3 cc. of lead nitrate solution diluted with 5 cc. of water, add 3 cc. of sodium iodide solution. *Write the equation for the reaction.* Heat the solution to boiling. *What happens?* Cool the solution again. *What property of lead iodide is indicated?*

d. Repeat (c) using potassium dichromate ($K_2Cr_2O_7$) solution instead of potassium iodide. *Name and describe the precipitate. Write the equation for its formation.*

e. To 3 cc. of lead nitrate solution diluted with 5 cc. of water, add 10 drops of dilute sulfuric acid. *Name and describe the precipitate. What other insoluble sulfate have you prepared?*

***Summarize** the way in which you would use these experiments to identify compounds of lead in solution. Which of these experiments is the more delicate test for lead?*

EXPERIMENT 81

Copper Compounds

Apparatus. — Test tubes and rack. Small flask. Tripod. Asbestos gauze. Bunsen burner.

Materials. — Crystals of copper sulfate, sodium chloride, copper chloride, and sodium sulfate. Copper filings. Concentrated hydrochloric acid. Sodium hydroxide solution. Litmus papers. Potassium ferrocyanide solution. Concentrated ammonium hydroxide.

Color of cupric ions. Dissolve in separate test tubes small crystals of copper sulfate, copper chloride, sodium chloride, and sodium sulfate in 15 cc. of water. *Note the color of the solutions. What are the colors, respectively, of the cupric* (Cu^{++}), *sodium* (Na^+), *sulfate* (SO_4^{--}), *and chloride* (Cl^-) *ions?*

Cuprous compounds. Dissolve a teaspoonful of cupric chloride ($CuCl_2$) in 15 cc. of water in a small flask. Add a teaspoonful of copper filings and 5 cc. of concentrated hydrochloric acid, and boil gently until the green color has quite disappeared. Pour half of the product into 100 cc. of water. *What is the color of the cuprous ions* (Cu^+)? *Write the equation for the reduction of cupric salts with copper.*

To the remainder of the cuprous chloride ($CuCl$) solution add sodium hydroxide solution until the solution is strongly alkaline. *Note the color of the precipitate.* Try the effect of heating it. *Write the equations representing these changes.*

Cupric compounds. Test a solution of copper sulfate with litmus paper. Explain the reaction by means of the ionic theory. (See p. 344 in B. & C., NEW PRACTICAL CHEMISTRY).

Add sodium hydroxide solution to a cold solution of copper sulfate as long as it forms a precipitate. *Note the color of the precipitate.* Heat the mixture until no further change takes place. *Write the equations for these changes.*

Tests for cupric ions (Cu^{++}). **a.** Add a few drops of copper sulfate solution to half a test-tubeful of water and then add potassium ferrocyanide ($K_4Fe(CN)_6$) solution. The precipitate is cupric ferrocyanide ($Cu_2Fe(CN)_6$). *Write the equation for this reaction.*

b. Another characteristic test for the cupric ion is to add concentrated ammonium hydroxide drop by drop to the copper solution until no further change is produced. The deep-blue color is due to a complex copper-ammonia ion ($Cu(NH_3)_4^{++}$).

EXPERIMENT 82

Analysis of the Oxides of Copper*

(*Quantitative Experiment*)

Apparatus. — Combustion tube, Pyrex resistance glass (30 cm. × 13 mm. inside diam.). Combustion boat, porcelain (6 cm. × 7 mm.). Corks. Glass tubing. Bunsen burner or preferably Fisher form of Méker. Horn-pan balance and weights. Ring stand and clamp.

Materials. — Cupric oxide, black powder, c.p. Cuprous oxide, red powder, c.p.

Introduction. We may demonstrate the truth of the Law of Multiple Proportions by analyzing the oxides of copper. When these oxides are heated in a current of hydrogen, metallic copper is left. The oxygen has united with the hydrogen to produce water. Since coal gas is largely made up of hydrogen, it can be used as the reducing agent. The loss in weight which takes place when the oxide is reduced gives the weight of oxygen in the oxide. Then we can calculate the percentage composition of each oxide. Assuming the atomic weights of copper and oxygen, we can compute the simplest formula for each oxide.

Directions. **a.** ***Cupric oxide.*** The essential part of the apparatus used in this experiment is shown in figure 96. A porcelain boat B is weighed and then weighed again with black copper oxide placed within it. The boat is then slipped into the hard-glass tube T, which has been provided with a cork at each end carrying a glass tube. The tube C is connected to the gas supply, and the tube O is bent upwards and constricted at its outer end to form a jet. The hard-glass tube T is clamped near one end so that the end carrying the tube O is slightly lower than the end carrying the tube C. The purpose of this tilt is to keep any drops of water which may be formed from running back on the hotter part of the tube T and so cracking it.

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* This experiment may well be carried out as a *Coöperative Experiment,* half of the class doing part (a) and the rest part (b).

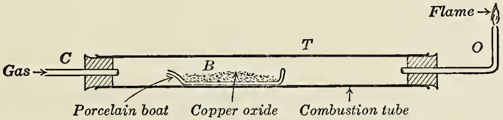

Fig. 96. Reduction tube and porcelain boat.

Test the apparatus in order to be sure that it is air-tight. Dry some pulverized cupric oxide by heating it in a porcelain crucible and then allowing it to cool in a desiccator. Use about 1.5 grams of the cupric oxide.

Allow the gas to pass through tube T for a minute and test the gas coming from tube O to make sure that all the air has been displaced. Then light the gas as it comes out of the jet O. Begin heating tube T, very gently at first, under one end of the boat B. You will soon notice that the black color of the cupric oxide at that end of the boat is changing to red (metallic copper). As this change takes place, move the flame gradually so as to heat the rest of the boat until all the black color is gone. Allow the tube to cool with the gas still passing through. When the tube is cold, the boat is removed and reweighed.

The data and calculations should be put into your notebook in tabular form:

Data:

Weight of boat and black cupric oxide	g.
Weight of boat	g.
Weight of black cupric oxide	g.
Weight of boat and copper	g.
Weight of boat	g.
Weight of copper	g.

Calculation:

Weight of cupric oxide . . . . g.
Weight of copper . . . . g.
Weight of oxygen . . . . g.
Percentage of oxygen in cupric oxide . . . . %
Percentage of copper in cupric oxide . . . . %

$$\frac{\text{Percentage of oxygen}}{16} = \ldots\ldots$$

$$\frac{\text{Percentage of copper}}{63.6} = \ldots\ldots$$

Therefore the simplest formula for cupric oxide is -----.

b. *Cuprous oxide.* In the meantime another student will repeat this experiment, using about 2.5 grams of cuprous oxide. Pure cuprous oxide is a red powder which tends to pass into the cupric oxide. It can be kept successfully if sealed up in a small bottle which is not opened until required. If the bottle has been opened for some time, its contents should not be used in this experiment.

EXPERIMENT 83

Borax Bead Tests for Metals

Apparatus. — Platinum (mounted). Bunsen burner.
Materials. — Borax (powdered). Cobalt nitrate. Manganese dioxide. Chromium sulfate. Ferrous sulfate. Nickel nitrate. Unknowns.

Introduction. Ordinary borax is hydrated sodium tetraborate ($Na_2B_4O_7 \cdot 10\,H_2O$). When heated, borax swells because of the evaporation of the water of hydration and then melts to a clear glassy mass. The oxides of certain metals impart characteristic colors to beads of borax, which acts as a flux dissolving the metallic oxide.

Making borax beads. Attach one end of a piece of platinum wire to a piece of glass tubing by melting the glass. Bend the other end of the platinum wire into a loop, about 3 mm. in diameter. Heat the wire in the Bunsen flame and while still hot dip it quickly into some powdered borax. Heat strongly in the flame. After a moment the borax shrinks into a small transparent bead. Dip this again into the borax and repeat this process until you have formed a round bead as large as the loop.

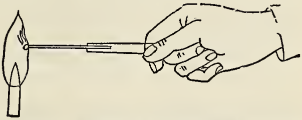
Fig. 97. Making a borax bead in the outer flame.

Tests for metals. Touch this borax bead, while still hot, to a tiny bit of some cobalt compound, such as cobalt nitrate. Heat again in the hot outer portion (Fig. 97) of the Bunsen flame (oxidizing) until the color is uniform. If the bead is black, too much of the salt was used. Try again. To remove the bead, dip it, while red-hot, in water. *Record in your notebook the characteristic colors:*

METALS	FORMULA OF COMPOUND	COLOR OF BEAD IN OXIDIZING FLAME
Cobalt, etc.		

Repeat this experiment with compounds of **manganese, chromium, iron,** and **nickel,** using each time a newly made bead. Learn the characteristic colors of these metals.

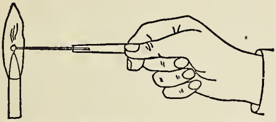
Fig. 98. Making a borax bead in the inner flame.

Repeat this experiment using the reducing flame, *i.e.* just at the tip of the inner cone (Fig. 98), with each metal. Also note any differences in color between the bead when hot and when cold.

Test for an unknown. Ask the instructor for an unknown salt to test by the bead method.

EXPERIMENT 84

Silver Compounds in Photography

Apparatus. — Test tubes and rack. Funnel. Shallow dish or tray.

Materials. — Silver nitrate solution. Potassium bromide solution. Filter papers. Tube of photographic developing powder. Sodium thiosulfate (hypo) solution. Potassium chloride solution.

Introduction. The sensitive plate or film is coated with silver bromide ($AgBr$) embedded in gelatin. After exposure to light (the eye detects no changes), the plate is placed in a developing solution. This solution contains various carbon compounds derived from benzene, such as pyrogallol, hydroquinone, and metol, and acts upon the silver bromide in proportion to the intensity of the light falling on the different parts of the plate. The silver bromide is reduced to metallic silver, and thus the image is formed. As soon as the image has acquired sufficient density, the plate is washed and placed in a solution of sodium thiosulfate (hypo), which removes all the silver bromide that the light and developing solution have not affected. Since no further change can take place on the plate, the image is said to be "fixed" and the "negative" is made.

This experiment should be done in a darkened room or in test tubes wrapped in dark paper.

Directions. **a.** ***Action of light on silver bromide.*** Add 5 cc. of silver nitrate solution to 5 cc. of potassium bromide solution. *Describe the precipitate and write the equation for its formation.* Pour a little of the precipitate on a filter paper and place it in the direct sunlight. *Examine it after a few minutes.*

Expose the precipitate remaining in the test tube to a bright light for a few seconds and then add 5 cc. of a developing solution. Allow the action to continue for 2–3 minutes. *Describe any changes occurring in the precipitate.* Then add 5 cc. of a saturated solution of sodium thiosulfate (hypo) and shake the mixture. *What is the effect of adding the thiosulfate?*

b. ***Silver bromide, not exposed to light.*** Add 5 cc. of silver nitrate solution to 5 cc. of potassium bromide solution, but without exposing to the light the silver bromide formed. Then add 5 cc. of the developer. Keep it in the dark for a few minutes. *What is the result?* Add 5 cc. of the "hypo" solution. *Compare the result with that obtained in* (a).

c. ***Printing.*** Pour a little silver nitrate solution into a shallow dish and float upon it a piece of well-sized paper. Remove the paper and let it dry. Then float it on the surface of a little potassium chloride solution in another dish. Again dry the paper. All this must be done in a darkened room or closet. Place a key or opaque stencil over the paper and expose it to the light for a few minutes. Examine it in a dim light and then immerse the paper in a developing solution for 2 minutes. Rinse it and place it in the "hypo" solution for 10 minutes. Rinse it thoroughly; dry and mount it in your notebook.

EXPERIMENT 85

Separation of Silver, Mercury, and Lead

Apparatus. — Test tubes and rack. Funnel. Bunsen burner.

Materials. — Solutions of silver, lead, and mercurous nitrates. Hydrochloric acid, concentrated and dilute. Nitric acid, concentrated and dilute. Concentrated and dilute ammonium hydroxide. Solution of potassium chromate. Copper strip. Filter papers. Litmus papers. Unknown solutions.

Silver chloride ($AgCl$). Add to 5 cc. of silver nitrate ($AgNO_3$) solution an equal volume of water and then add drop by drop dilute hydrochloric acid. Shake between the drops and allow the precipitate to settle until the next drop produces no further precipitation. *What is the precipitate? Write the equation.*

Let the precipitate settle and then pour away the liquid. *What does it contain?* Add to the precipitate a little distilled water and shake the mixture. Allow the precipitate to settle and again pour off the liquid. Now try to dissolve the silver chloride in boiling hot water. *Is silver chloride soluble in hot water?*

Divide the remaining silver chloride into two parts. To one part add dilute ammonium hydroxide. *Is silver chloride soluble in ammonium hydroxide?* Set the other part of the silver chloride in the sunlight and examine it later.

Mercurous chloride ($HgCl$). To 5 cc. of mercurous nitrate ($HgNO_3$) solution, add 5 cc. of distilled water and then add dilute hydrochloric acid, drop by drop, until the action is complete. *Write the equation.* Wash the precipitate with cold water; then divide it into two parts. *Does mercurous chloride dissolve in hot water?* Add to the other portion of the precipitate some concentrated ammonium hydroxide. *How would you test for a soluble mercurous salt?*

Lead chloride ($PbCl_2$). Dilute 5 cc. of lead nitrate ($Pb(NO_3)_2$) solution with 5 cc. of distilled water and then add hydrochloric acid, drop by drop, until the precipitation is complete. *Name the precipitate and write the equation.* Wash the precipitate with cold water and boil it with distilled water. *Is lead chloride soluble in hot water?*

Divide the solution into two parts. To one part add a solution of potassium chromate (K_2CrO_4). *What is the precipitate? Write the equation.* Examine the other portion of the solution after it has become cold. *How would you test for a soluble lead salt?*

The separation. Make a mixture in one test tube of solutions of silver nitrate, lead nitrate, and mercurous nitrate. Add dilute hydrochloric acid until the precipitation is complete. Filter and wash the precipitated chlorides with a little *cold* water.

To separate the **lead**, wash the precipitate thoroughly with *boiling* water and keep the washings. *Which one of the chlorides will dissolve? Test the filtrate for the presence of this compound.*

Next, to separate the **silver**, wash the precipitate remaining on the filter paper with dilute ammonium hydroxide and keep the washings. *Which chloride is now dissolved? Which remains on the filter?* Add to the filtrate a slight excess of nitric acid (test with litmus paper). *Name the precipitate.*

Finally, to show that the residue left on the filter contains a compound of **mercury**, pour on it a little *aqua regia* (1 cc. of concentrated hydrochloric acid and 0.5 cc. of concentrated nitric acid) and catch the filtrate in a clean test tube. Dilute the solution thus obtained with 5 cc. of water and put into it a small strip of bright copper. After a few minutes, remove the copper; wash and rub it. *Explain the result.*

Testing an unknown. Obtain from the instructor a solution which contains one or more of these metals (silver, mercury, and lead) and analyze the solution. *Report the negative as well as the positive results.*

EXPERIMENT 86

Analysis of a Silver Coin

(*Quantitative Experiment*)

Apparatus. — Two beakers. Tripod. Asbestos gauze. Bunsen burner. Stirring rod. Funnel. Wash-bottle. Horn-pan balance and weights. Ring stand.

Materials. — Dime (cut into 2 or 3 pieces). Concentrated nitric acid. Dilute hydrochloric acid. Filter papers.

Introduction. When a dime is dissolved in nitric acid, the metals (silver and copper) are converted into nitrates. The silver, which is precipitated with hydrochloric acid as silver chloride, is filtered off, dried, and weighed. From the weight of the silver chloride, the weight of silver in the coin and the percentage of silver present may be calculated.

Directions. Weigh to a centigram (0.01 g.) a portion of a clean ten-cent piece, place it in a small beaker, and pour in about 20 cc. of nitric acid (diluted with an equal volume of water). As soon as the vigorous action is over, warm the solution gently and add more acid, if necessary, to dissolve the metal. Then dilute the solution by adding 30 cc. of distilled water.

Slowly add hydrochloric acid to the metallic nitrate solution until the precipitation is complete. Keep the solution hot, stirring vigorously, until the precipitate settles readily and leaves the liquid perfectly clear. To prove that the precipitation is complete, add another drop of hydrochloric acid. *How does this show that the precipitation is complete?*

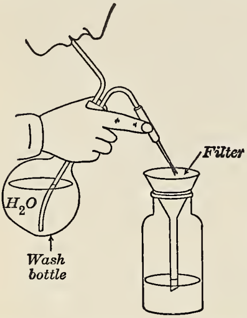

Fig. 99. Washing a precipitate with a wash-bottle.

Make ready in a funnel a *weighed* filter paper. Decant the clear liquid down a rod through the filter. Transfer *all* the precipitate to the filter. In order to get out the last particles, use a glass rod and a stream from the wash-bottle (Fig. 99). Wash the precipitate several times with distilled water and then set it aside to dry on the paper. It is well to leave the precipitate in a drying oven, where it will become thoroughly dry without burning the paper.

Get the weight of the silver chloride formed. From this and the approximate atomic weights (Table inside front cover) *calculate the weight of silver in a dime.* Finally, *compute the percentage of silver in the coin.*

Show all your computations and arrange your data and results in tabular form.

EXPERIMENT 87

Dyeing

(*Optional Experiment*)

Apparatus. — Beakers. Stirring rod.

Materials. — Concentrated solutions of picric acid and logwood. Strips of cotton cloth (1″ × 2″). Woolen yarn. Sodium carbonate solution (2%). Congo red. Sodium sulfate solution (10%).

Introduction. Nearly all the dyes used to-day are complex organic substances prepared from coal tar. Many dyestuffs are soluble in water, and when the cloth is placed in such highly colored solutions, the dye becomes attached to the cloth. Silk and wool are particularly easy to dye in this way. But there are many other colored substances which can be used as dyes only if employed in connection with a metallic hydroxide called a **mordant.** If the hydroxide is precipitated on the cloth in the presence of the dye, it will bind the colored material to the fabric. Because some dyes give different colors with different mordants, a pattern of colors may be dyed on a cloth in one dyeing.

Directions. a. ***Direct dyeing.*** Fill a beaker half full of a concentrated solution of picric acid. Put in it a piece of clean, white cotton cloth * and a few strands of white woolen yarn. Boil for about five minutes. Keep the cloth entirely immersed in the dye. Remove both the cotton and wool, and wash them thoroughly with water. *Compare the results. In which fabric is the color fast?*

Repeat the experiment, using logwood solution instead of picric acid.

b. ***Dyeing with a mordant.*** To mordant cotton cloth, soak it in a solution of aluminum sulfate and squeeze out the excess liquid. Now dip the cloth in ammonium hydroxide and again squeeze out the excess liquid. *What compound is now incorporated in the cloth? Write the equation for its formation.*

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* To remove all foreign matter from the cloth, place the strips in a beaker and cover them with sodium carbonate solution (2 per cent solution). Boil the liquid for 5 minutes. Remove the strips and rinse thoroughly with water.

Boil the mordanted cloth in logwood solution for about 5 minutes. Wash and dry the cloth. *Compare the color of the mordanted cotton dyed in logwood with the color of the unmordanted cotton dyed in logwood.*

When the pieces of cloth and yarn are dry, attach them (properly labeled) to your notebook.

c. *Dyeing without a mordant.* Some dyestuffs will dye cotton fast without the use of mordants. Congo red (sold at the druggist's) is a dyestuff of this class. Prepare a solution containing 2 g. sodium carbonate, 10 g. sodium sulfate, and 0.2 g. Congo red in 100 cc. of water. Heat the solution to boiling and immerse in it a wet unmordanted strip of cotton and continue boiling for 5 minutes. Remove the cloth, rinse, and test to see whether the cloth is dyed fast.

EXPERIMENT 88

Paints

(Optional Experiment)

Apparatus. — Test tubes. Funnel. Filter papers. Bunsen burner.

Materials. — Solutions of potassium iodide, mercuric chloride, potassium chromate (sat.), lead acetate (sat.), ferric chloride (25 per cent), potassium ferrocyanide (25 per cent), sodium hydroxide (10 per cent). Wood splints. Linseed oil. Turpentine. Absorbent cotton.

Introduction. A paint is a mixture of a **pigment**, a **vehicle**, or carrier, and a **drier**. A pigment is a finely divided, insoluble colored powder. One may judge the value of a pigment according to its *body*, *covering power*, *drying quality*, *durability*, and its *beauty of shade.* The vehicle is a fluid (such as linseed oil or pyroxylin) which acts as a carrier for the pigment and which oxidizes when exposed to air with the production of a hard, elastic surface. A drier increases the drying qualities of the paint. Oil paints "dry" because of oxidation; water colors, pyroxylin paints, and spirit varnishes (shellac) dry by evaporation of one of the components. In this experiment you will prepare a number of typical pigments.

Directions. a. *Brilliant scarlet.* Fill a test tube half full of a solution of potassium iodide. To this solution add a solution of mercuric chloride. Filter, wash, and dry the precipitate. *Name the product, describe its color, and write the equation for its formation.*

b. *Chrome yellow.* Fill a test tube half full of a concentrated solution of potassium chromate and add a solution of lead acetate. Shake the tube vigorously. Filter, wash, and dry the precipitate. *Name the product, describe its color, and write the equation for its formation.*

c. *Prussian blue.* Fill a test tube one-third full of ferric chloride solution and then add an equal volume of potassium ferrocyanide solution. Filter, wash, and dry the precipitate. *Note the color of the precipitate. Give its chemical name.*

d. *Chrome orange.* Prepare a second portion of lead chromate (b), pour off the liquid, and add to the precipitate about 5 cc. of a solution of sodium hydroxide. Boil the mixture gently for several minutes until the precipitate changes to an orange color. This pigment consists of a basic chromate of lead of rather variable composition dependent upon the length of time the action is allowed to proceed. Filter, wash, and dry the precipitate. *Note its color.*

e. *Brunswick green.* This pigment is prepared by mixing together in various proportions barytes ($BaSO_4$), Prussian blue, and chrome yellow. A series of shades varying from pale greenish-yellow to deep bluish-green may be prepared. On a clean filter paper mix a small portion of chrome yellow (b) with an equal portion of Prussian blue (c). *Record the color of the resulting pigment.*

f. To each residue left on the filter papers add a few drops of linseed oil. Mix well, using a clean splint for each pigment. Add a drop or two of turpentine to make a paint of the right consistency. With clean pieces of absorbent cotton smear a square (1″ on a side) in your notebook with each paint prepared. *Label each square with its pigment and allow it to dry.*

EXPERIMENT 89

Identification of Simple Compounds

Apparatus. — Test tubes. Ignition tube. Bunsen burner. Platinum wire. Charcoal block. Cobalt glass.

Materials. — Solutions of silver nitrate, barium chloride, cobalt nitrate, and ferrous sulfate. Dilute hydrochloric and nitric acids. Concentrated sulfuric acid. Borax. Unknowns.

Introduction. One of the most interesting and practical divisions of chemistry is **qualitative analysis,** by which one is able to determine what elements are present in materials. This part of chemistry has been developed to such a degree of precision that it is almost a special art and science by itself. In this experiment you will have a practical review of the tests which you have already studied for a number of metals and acids. You will be given a small sample of a material the composition of which is unknown to you. In making each test use only a very small portion, and always keep a little for verification of your results. Keep a record of all the *tests* you make and of the *results* even though they are negative. When you have decided what is in the unknown material, it is well to apply all the confirmatory tests which are appropriate.

Directions. **a.** If the material is a **solid,** it will be well to make a preliminary examination by means of (1) the *flame test* with clean platinum wire, (2) the *cobalt nitrate test,* and (3) the *borax-bead test.*

b. *Heat* the substance in a small ignition tube as hot as possible. Water in the tube may mean water of hydration or water held mechanically. Blackening indicates an organic compound. A colorless gas might indicate a carbonate, an ammonium compound, or a sulfite. Brown fumes indicate a nitrate, a nitrite, or a bromide. Violet fumes indicate an iodide. A sublimate indicates a mercury or an ammonium compound.

c. *Add dilute hydrochloric acid.* Effervescence with colorless gas indicates a carbonate, a sulfite, or a sulfide. Brown fumes indicate a nitrite.

d. *Add concentrated sulfuric acid.* A colorless gas indicates a chloride, sulfide, sulfite, carbonate, or acetate. Brown fumes indicate a bromide, nitrate, or nitrite. Purple fumes indicate an iodide.

e. If the unknown material is wholly or partially soluble in water, test the water solution for a chloride, a sulfate, and a nitrate.

f. *Add hydrochloric acid.* A precipitate indicates silver, mercury (-ous), or lead, which may be separated as in Exp. 85.

g. *Bubble hydrogen sulfide* into the liquid made slightly acid with hydrochloric acid. A yellow precipitate indicates cadmium or tin (-ic). A black precipitate indicates copper, mercury (-ic), or lead. A brown precipitate indicates tin (-ous).

h. Boil the solution and make it alkaline with ammonium hydroxide. Then *add ammonium sulfide.* A black precipitate indicates iron, cobalt, or nickel. A white precipitate indicates zinc or aluminum. A pink precipitate indicates manganese.

i. Boil the solution and *add ammonium chloride* and afterwards *ammonium carbonate* solution. A white precipitate indicates barium, strontium, or calcium. Try flame tests.

j. After the above metals have been removed or proved absent, the solution may still contain magnesium, sodium, potassium, and ammonium. To some of the solution *add sodium phosphate* (Na_2HPO_4). A white crystalline precipitate indicates magnesium. Sodium and potassium can easily be detected with the flame test. Ammonium compounds give the odor of ammonia when heated with sodium hydroxide.

After one has identified any of the above metals, it is well to look up the special properties of its compounds and to apply some confirmatory tests.*

Don't fail to record the number of your unknown and to state what you decide that it contains, with your reasons.

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* It will be well at this point to consult some textbook on qualitative analysis, such as A. A. Noyes's *Qualitative Chemical Analysis*, published by The Macmillan Company.

EXPERIMENT 90

Inorganic Chemical Preparations

Introduction. The work of the chemist consists in preparing substances in a purer form than was available hitherto, or by a cheaper method. It will be interesting to learn how to prepare and purify substances. In an elementary course we must limit ourselves in the main to some of the simpler inorganic substances. We suggest that you select some element or compound in which you are interested and that you read up in your school-library reference books how it may be prepared in the laboratory. You will naturally have to confine yourself to such chemicals and apparatus as are found in your school laboratory, or to such as are available in the neighboring drugstore.

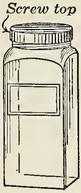

Fig. 100. Specimen bottle.

General directions. Among the processes which you may need to use are *solution*, *precipitation*, *filtration*, *decantation*, *washing*, *evaporation*, *crystallization*, *drying*, and *pulverizing*. Be sure to plan ahead carefully and to submit to your instructor a detailed, written description of your proposed method of procedure for making the preparation. Record accurately the weights of materials to be used, and calculate the amount of the product which you expect to make. And finally, weigh the actual substance that you obtain and hand it in with your report, neatly labeled in a clean, dry bottle (Fig. 100). Include in your report careful descriptions of the tests which you have applied to the finished product in order to prove that it is actually what you expected to get.

The following materials are suggested as possibilities:

1. Chrome alum ($K_2SO_4 \cdot Cr_2(SO_4)_3 \cdot 24\ H_2O$)
2. Microcosmic salt ($HNaNH_4PO_4 \cdot 4\ H_2O$)
3. Tartar emetic ($KSbO(C_4H_4O_6) \cdot \frac{1}{2}\ H_2O$)
4. Blue vitriol ($CuSO_4 \cdot 5\ H_2O$)

5. Copperas ($FeSO_4 \cdot 7\ H_2O$)
6. Lead chromate ($PbCrO_4$)
7. Epsom salts ($MgSO_4 \cdot 7\ H_2O$)
8. Vermilion (HgS)
9. Rochelle salts ($KNaC_4H_4O_6 \cdot 4\ H_2O$)
10. Zinc sulfate ($ZnSO_4 \cdot 7\ H_2O$)

NOTE. Do not begin work on your preparation until your plan has been approved by your instructor. Naturally he will not consent to your working with dangerous substances or expensive and elaborate apparatus. It is expected that you will devote your spare time for about a month to this experiment. In grading your results, account will be taken of the initiative, energy, and skill displayed as well as the quantity and quality of the product.

APPENDIX

PRESSURE OF WATER VAPOR, OR AQUEOUS TENSION

(In millimeters of mercury)

Temperature	Pressure	Temperature	Pressure	Temperature	Pressure
0° C.	4.6 mm.	11° C.	9.8 mm.	22° C.	19.8 mm.
1°	4.9	12°	10.5	23°	21.0
2°	5.3	13°	11.2	24°	22.3
3°	5.7	14°	12.0	25°	23.7
4°	6.1	15°	12.8	26°	25.1
5°	6.5	16°	13.6	27°	26.7
6°	7.0	17°	14.5	28°	28.3
7°	7.5	18°	15.5	29°	29.9
8°	8.0	19°	16.5	30°	31.7
9°	8.6	20°	17.5	50°	92.3
10°	9.2	21°	18.6	100°	760.0

DENSITIES OF IMPORTANT GASES

(Approximate weight of 1 liter under standard conditions)

Acetylene (C_2H_2)	1.16 grams
Air	1.29 "
Ammonia (NH_3)	0.77 "
Carbon dioxide (CO_2)	1.98 "
Carbon monoxide (CO)	1.25 "
Chlorine (Cl_2)	3.17 "
Helium (He)	0.18 "
Hydrogen (H_2)	0.09 "
Hydrogen chloride (HCl)	1.64 "
Hydrogen sulfide (H_2S)	1.54 "
Methane (CH_4)	0.72 "
Nitric oxide (NO)	1.34 "
Nitrogen (N_2)	1.25 "
Nitrous oxide (N_2O)	1.98 "
Oxygen (O_2)	1.43 "
Sulfur dioxide (SO_2)	2.93 "

DENSITY OF HYDROGEN (in grams per liter), MEASURED OVER WATER, AT VARYING TEMPERATURES AND PRESSURES.

(To get weight of 1 cc., move decimal point 3 places to left.)

PRESSURE (mm.)	TEMPERATURE IN DEGREES CENTIGRADE									
	15°	16°	17°	18°	19°	20°	21°	22°	23°	24°
740	0.0815	0.0811	0.0807	0.0803	0.0799	0.0795	0.0792	0.0787	0.0784	0.0780
741	.0816	.0812	.0808	.0804	.0800	.0797	.0793	.0788	.0785	.0781
742	.0817	.0813	.0809	.0805	.0801	.0798	.0794	.0789	.0786	.0782
743	.0818	.0814	.0810	.0806	.0803	.0799	.0795	.0790	.0787	.0783
744	.0819	.0815	.0812	.0808	.0804	.0800	.0796	.0792	.0788	.0784
745	.0820	.0817	.0813	.0809	.0805	.0801	.0797	.0793	.0789	.0785
746	.0821	.0818	.0814	.0810	.0806	.0802	.0798	.0794	.0790	.0786
747	.0823	.0819	.0815	.0811	.0807	.0803	.0799	.0795	.0791	.0787
748	.0824	.0820	.0816	.0812	.0808	.0804	.0800	.0796	.0792	.0788
749	.0825	.0821	.0817	.0813	.0809	.0806	.0802	.0797	.0793	.0789
750	.0826	.0822	.0818	.0814	.0810	.0807	.0803	.0798	.0795	.0791
751	.0827	.0823	.0819	.0815	.0811	.0808	.0804	.0799	.0796	.0792
752	.0828	.0824	.0820	.0816	.0813	.0809	.0805	.0800	.0797	.0793
753	.0829	.0826	.0822	.0818	.0814	.0810	.0806	.0802	.0798	.0794
754	.0830	.0827	.0823	.0819	.0815	.0811	.0807	.0803	.0799	.0795
755	.0832	.0828	.0824	.0820	.0816	.0812	.0808	.0804	.0800	.0796
756	.0833	.0829	.0825	.0821	.0817	.0813	.0809	.0805	.0801	.0797
757	.0834	.0830	.0826	.0822	.0818	.0814	.0810	.0806	.0802	.0798
758	.0835	.0831	.0827	.0823	.0819	.0815	.0812	.0807	.0803	.0799
759	.0836	.0832	.0828	.0824	.0820	.0817	.0813	.0808	.0804	.0800
760	.0837	.0833	.0829	.0825	.0822	.0818	.0814	.0809	.0806	.0802
761	.0838	.0835	.0831	.0826	.0823	.0819	.0815	.0810	.0807	.0803
762	.0839	.0836	.0832	.0828	.0824	.0820	.0816	.0812	.0808	.0804
763	.0841	.0837	.0833	.0829	.0825	.0821	.0817	.0813	.0809	.0805
764	.0842	.0838	.0834	.0830	.0826	.0822	.0818	.0814	.0810	.0806
765	.0843	.0839	.0835	.0831	.0827	.0823	.0819	.0815	.0811	.0807
766	.0844	.0840	.0836	.0832	.0828	.0824	.0820	.0816	.0812	.0808
767	.0845	.0841	.0837	.0833	.0829	.0825	.0822	.0817	.0813	.0809
768	.0846	.0842	.0838	.0834	.0830	.0826	.0823	.0818	.0814	.0810
769	.0847	.0844	.0840	.0835	.0832	.0828	.0824	.0819	.0815	.0812

THE METRIC SYSTEM

In this system of weights and measures the relation of the units is expressed in multiples of **ten.** During the last hundred years most of the civilized nations of the world, except Great Britain and the United States, have adopted the **metric system.** Even in these countries it is almost universally used in scientific work; and in the United States, fortunately, the system of money is made on the decimal plan.

Some important units of length. The unit is the **meter,** which is equal to 39.37 inches.

1 meter (m.) = 100 centimeters (cm.).
1 centimeter = 10 millimeters (mm.).
1 kilometer (km.) = 1000 meters.
2.54 centimeters = 1 inch.

The chemist needs to become familiar with the centimeter and millimeter. Thus, the barometer may read 760 mm. or 76 cm. or about 30 inches.

Units of volume or **capacity.** The unit of volume that is most extensively used in the laboratory is the volume of a cube of which the edge is 1 centimeter. This is called a **cubic centimeter** (cc.).

1 liter (l.) = 1000 cubic centimeters (cc. or ml.).
1 liter = 1.06 quarts.

Thus, a 500-cc. flask holds half a liter or about a pint (16 ounces of water).

Units of Weight. The unit of weight is the **gram,** which is approximately the weight of one cubic centimeter of water.

1 gram (g.) = 1000 milligrams (mg.).
1 gram = 100 centigrams (cg.).
1 kilogram (kg.) = 1000 grams.
1 kilogram = 2.2 pounds.
1 ounce (avoirdupois) = 28.35 grams.

The chemist usually expresses weights in grams and decimal fractions thereof. Thus, a crucible may weigh 12.53 g. Every student in chemistry needs to become very familiar with **centimeters, cubic centimeters,** and **grams.**

TABLE OF SOLUBILITIES

S, soluble in water. P, slightly soluble in water.
I, insoluble in water. Ia, insoluble in water and dilute acids.

	Acetate	Bromide	Carbonate	Chloride	Hydroxide	Iodide	Nitrate	Oxide	Phosphate	Sulfate	Sulfide
Aluminum	S	S	—	S	I	S	S	I	I	S	—
Ammonium	S	S	S	S	S	S	S	—	S	S	S
Barium	S	S	I	S	P	S	S	—	I	Ia	S
Calcium	S	S	I	S	P	S	S	—	I	P	P
Copper	S	S	I	S	I	S	S	I	I	S	Ia
Ferrous (Fe^{++})	S	S	I	S	I	S	S	I	I	S	I
Ferric (Fe^{+++})	—	S	—	S	I	—	S	I	I	S	—
Lead	S	P	I	P	I	I	S	I	I	Ia	Ia
Magnesium	S	S	I	S	I	S	S	I	I	S	I
Manganese	S	S	I	S	I	S	S	I	P	S	I
Mercurous (Hg^{+})	P	I	I	I	—	I	S	I	I	P	Ia
Mercuric (Hg^{++})	S	S	I	S	I	I	S	I	I	S	Ia
Nickel	S	S	I	S	I	S	S	I	I	S	I
Potassium	S	S	S	S	S	S	S	S	S	S	S
Silver	S	Ia	I	Ia	—	Ia	S	I	I	P	Ia
Sodium	S	S	S	S	S	S	S	S	S	S	S
Stannous (Sn^{++})	S	S	—	S	I	S	—	I	I	S	Ia
Stannic (Sn^{++++})	S	S	—	S	I	S	—	I	I	—	Ia
Zinc	S	S	I	S	I	S	S	I	I	S	I

GENERAL RULES FOR SOLUBILITY

(The exceptions to these rules are few and unimportant.)

1. All sodium, potassium, and ammonium compounds are soluble in water.
2. All nitrates, chlorates, and acetates are soluble in water.
3. All chlorides are soluble, except those of silver, mercury (mercurous), and lead (lead slightly soluble).
4. All sulfates are soluble, except those of barium, lead, and calcium (calcium slightly soluble). The silver and the mercurous sulfates are only moderately soluble.
5. All carbonates are insoluble, except those of sodium, potassium, and ammonium.
6. All oxides and hydroxides are insoluble, except those of ammonium, sodium, potassium, and barium (calcium hydroxide slightly soluble).

EXPERIMENTS AND CORRESPONDING CHAPTERS

The Arabic numerals refer to Black's *New Laboratory Experiments in Practical Chemistry*, and the Roman numerals to Black and Conant's *New Practical Chemistry.*

Experiments	Chapters	Experiments	Chapters
A, *B*	I	37, 38	XIX
1, 2	II	39, 40	XX
3, 4	III	41, 42, 43	XXI
5, 6	IV	—	XXII
7, 8	V	44, 45, 46	XXIII
9, 10	VI	47, 48, 49, 50	XXIV
11	VII	51, 52, 53	XXV
12	VIII	54, 55, 56, 57, 58, 59	XXVI
13	IX	60, 61, 62	XXVII
14, 15	X	63, 64	XXVIII
16, 17, 18	XI	65, 66	XXIX
19, 20	XII	67, 68, 69, 70, 71	XXX
21, 22, 23	XIII	72, 73, 74, 75	XXXI
—	XIV	76, 77, 78	XXXII
24, 25	XV	79, 80, 81, 82	XXXIII
26, 27	XVI	83, 84, 85	XXXIV
28, 29, 30, 31, 32	XVII	87, 88	XXXV
33, 34, 35, 36	XVIII	89, 90	XXXVI

LIST OF LABORATORY SUPPLIES

APPARATUS — INDIVIDUAL

Beakers (3), 100 cc., 250 cc., 400 cc.
Blowpipe, brass, 8″
Bottle, narrow neck, 1000 cc.
Bottles (4), wide mouth, 250 cc.
Brush, test-tube
Burner, Bunsen
Burner, Fisher form of Méker
Clamp, wire form for test tube
Clamps (2), iron, small; for burettes; for test tubes; etc.
Crucible, porcelain, with cover, # 0
Crucible, sheet-iron, with cover, 50 cc.
Dish, crystallizing, 3″ diam.
Dish, evaporating, porcelain, # 1
Dish, tin or iron, sand-bath
Dropper, medicine
File, triangular, 5″
Filter papers (25), diam. about 10 cm.
Flask, distilling with side tube, 250 cc.
Flask, Erlenmeyer, 125 cc.
Flask, Erlenmeyer, 250 cc.
Flask, Florence, 500 cc.
Forceps, brass, curved for weights
Forceps, iron, 3″
Funnel, 60°, 2½″
Gauze, iron with asbestos center, 5″ × 5″
Graduate, cylindrical, 100 cc.
Mortar and pestle, porcelain, about 7.5 cm.
Pan, agate, shallow, 1 qt.
Pinchcock (Hoffman's screw)
Plates, glass (4), 3″ × 3″
Platinum wire, # 28, 2″ (or Nichrome wire 6″)
Rack for 12 test tubes
Reagent bottles (H_2SO_4, HCl, HNO_3, NH_4OH), 4 oz., glass stopper, raised letters.
Retort, tubulated, with ground-glass stopper, 4 oz.
Rods (2), glass for stirring, 15 cm. long and 5 mm. diam.
Sponge
Spoon, combustion or deflagrating, bowl 1 cm.
Stand, iron, with 3 rings
Stoppers, rubber with one hole, (2) to fit test tube, (1) to fit ignition tube, (2) to fit combustion tubing, (1) to fit 250-cc. flask

Stoppers, rubber with two holes (1) to fit 500-cc. flask, (2) to fit wide-mouth bottle, (1) to fit large test tube, (1) to fit 250-cc. flask
Triangle, pipe-stem, size to support # 0 crucible
Tripod, iron, to support dishes over burner
Trough, pneumatic, with shelf or support for bottles
Tube, straight calcium chloride, with bulb, 6″
Tube, ignition (Pyrex), 6″ × $\frac{3}{4}$″
Tubes (12), test (soft glass), 6″ × $\frac{3}{4}$″
Tube, test (soft glass), 8″ × 1″
Tube, test, graduated, 25 cc.
Tube, thistle, stem 25 cm. × 6 mm.
Tubing, rubber, 2′, $\frac{1}{4}$″ in diam.
Tubing, rubber, 6″, $\frac{3}{16}$ ″ in diam.
Watch glasses (3), 2$\frac{1}{2}$″ in diam.
Wing-top for Bunsen burner

Apparatus — General. (*For 10 Students*)

5 Balances,* horn-pan, 7$\frac{1}{2}$″ beam, sensitive to 0.01 g.
2 Balances, trip scales with rider (0–10 g.) and weights (1 g. to 500 g.) in block
1 Barometer, mercury
5 Battery jars (4″ × 5″)
1 lb. Beads, glass, small
1 Blast lamp
1 Blower, rotary with electric motor; or foot bellows
2 Bottles, acid, about 2$\frac{1}{2}$ liters
4 Burettes, 50 cc. graduated to 0.1 cc., Mohr with fittings
6 Cells, dry or 2 six-volt storage batteries
2 pkg. Cigar lighters, wood splints
10 Cobalt-glass plates, double thickness, 2″ × 2″
5 Condensers, Liebig or spiral, 15″
5 Large clamps for ring stand and 6 ft. rubber tubing ($\frac{1}{4}$″) for connections for condensers
5 Combustion tubes, 20 cm. long, 15 mm. inside diam.
1 set Cork-borers (6) and sharpener
100 Corks, best grade, long, assorted sizes (# 7, 8, 9, 10, 12)
5 Crucibles, Dixon (sand), 4″ deep
1 Cylinder, graduated, 250 cc.
1 Cylinder, graduated, 500 cc.

* Some teachers may prefer to use a better balance (Fig. 11) with agate planes and agate knife edges, with three rider beams. Under these conditions 5 balances, if used in rotation, might serve 10 students, and separate sets of weights are not required.

2 Hoffman electrolysis App. (Fig. 31)
5 Electrolysis App. (Fig. 32)
2 Files, round
1 Fire extinguisher (carbon tetrachloride)
1 First-aid cabinet with emergency supplies
5 Gas-measuring tubes, 50 cc. graduated to $\frac{1}{10}$ cc.
1 Glass cutter
1 Hydrometer for light liquids
1 doz. Jars, pint, fruit
1 Kipp's generator, 1 qt.
2 Magnifying lenses (doublet)
1 Meter stick
1 Microscope, compound (1″ eyepiece, $\frac{2}{3}$″ and $\frac{1}{6}$″ objectives)
1 Milk-testing outfit (Babcock) for butter fat
1 Oven, drying, electric or gas
5 Pans, iron, 5″ shallow form " sand bath "
5 Pipettes, 10 cc., volumetric
5 Plates, glass, 4″ × 4″
1 Pliers, side-cutting
1 doz. Rules, wood, metric, 30 cm.
5 sheets Sandpaper # 1
1 pr. Shears, metal-cutting
1 pk. Splints, wood
1 pkg. Tapers, wax
5 Thermometers, graduated from − 10° to + 110° C.
5 lb. Tubing, soft glass, assorted, 6–7 mm. outside diameter
5 Waterbaths, copper, 5″
1 Waterstill, automatic, gas (Fig. 48 in B. & C., *New Practical Chemistry*)
5 sets Weights,* good grade, 50 g. to 0.01 g., fractional weights, all in covered wooden box

MATERIALS. (*For 10 Students*)

1 lb. Acid, acetic (30%) c. p.
1 lb. Acid, formic (50%)
12 lb. Acid, hydrochloric, c. p. (sp. gr. 1.19)
7 lb. Acid, nitric, c. p. (sp. gr. 1.42)
8 oz. Acid, oxalic, cryst., c. p.
18 lb. Acid, sulfuric, c. p. (sp. gr. 1.84)
1 gal. Alcohol, denatured
2 qt. Alcohol, ethyl (95%)
1 lb. Alum, potassium and aluminum sulfate
4 oz. Aluminum, sheet, 18 gauge

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* See footnote in regard to balances.

8 oz. Aluminum sulfate, cryst.
1 lb. Aluminum wire (# 18)
1 lb. Ammonium chloride, c. p.
10 lb. Ammonium hydroxide, c. p. (sp. gr. 0.90)
1 oz. Ammonium molybdate, cryst., c. p.
8 oz. Ammonium nitrate, cryst., c. p.
4 oz. Ammonium oxalate, cryst., c. p.
1 lb. Ammonium sulfide solution
4 oz. Antimony, powdered
3 sq. ft. Asbestos paper, $\frac{1}{32}''$ thick
1 lb. Barium chloride, cryst., c. p.
4 oz. Bismuth, metal (gran.)
1 lb. Bleaching powder (chloride of lime)
1 lb. Boneblack
1 lb. Borax powder
1 oz. Bromine, c. p.
4 oz. Cadmium, metal (mossy)
1 oz. Cadmium nitrate, c. p.
1 lb. Calcium carbide
5 lb. Calcium carbonate (marble chips)
1 lb. Calcium carbonate (precipitated chalk)
4 oz. Calcium chloride, cryst., c. p.
2 lb. Calcium chloride (fused or granular)
2 lb. Calcium oxide (quicklime) in tin can
1 lb. Calcium sulfate, gypsum
5 lb. Calcium sulfate, plaster of Paris
12 Carbon rods ($6'' \times \frac{3}{16}''$)
2 lb. Carbon bisulfide (technical)
1 lb. Carbon tetrachloride (technical)
12 Charcoal blocks for blowpiping
1 lb. Charcoal, wood (powdered)
4 oz. Chromium (ic) sulfate, dry, purified
1 oz. Cobalt chloride, c. p.
1 oz. Cobalt nitrate, c. p.
1 oz. Cochineal, powder
32 sq. in. Copper gauze, 80 mesh
8 oz. Copper sheet, 0.005'' thick
2 lb. Copper turnings, clean, fine
1 lb. spool Copper wire, # 18
1 lb. spool Copper wire, # 24
1 lb. spool Copper wire, # 30
1 lb. Copper chloride, c. p.
4 oz. Copper (ic) nitrate, cryst., c. p.

4 oz. Copper (ic) oxide, black powder, c. p.
4 oz. Copper (ous) oxide, red powder, c. p.
8 oz. Copper oxide, wire form
4 oz. Copper sulfate, anhydrous, c. p.
1 lb. Copper sulfate, cryst., c. p.
4 oz. Cotton, absorbent
1 lb. Ether, U. S. P. Squibb's
4 oz. Fehling's solution, two solutions in separate bottles
1 lb. Formaldehyde (37%)
10 g. Fuchsin (Rosaniline)
4 oz. Gelatin, gran.
1 oz. Glass wool, fine threads
1 lb. Glucose (sirup)
8 oz. Glycerin, white, U. S. P.
1 lb. Hydrogen peroxide, U. S. P.
1 oz. Iodine, resublimed, U. S. P.
4 oz. Iron chloride (ferric), c. p.
1 lb. Iron filings, clean, fine, or steel wool
8 oz. Iron powder reduced by hydrogen
1 lb. Iron (ferric) oxide, technical
1 lb. Iron pyrite
1 lb. Iron sulfate (ferrous), cryst., c. p.
1 lb. Iron sulfide (ferrous), sticks, for H_2S
1 pkg. Junket (rennet) tablets
1 lb. Kaolin (china clay)
1 lb. Lead, metal, foil (tea lead)
1 lb. Lead, metal, finely divided
4 oz. Lead acetate, cryst., c. p.
8 oz. Lead carbonate (basic, dry)
1 lb. Lead nitrate, cryst., c. p.
1 lb. Lead oxide (litharge), powder
1 lb. Lead sulfide (galena)
1 oz. Lithium chloride, c. p.
1 oz. Litmus cubes
2 tubes Litmus paper — 100 strips blue
2 tubes Litmus paper — 100 strips red
4 oz. Logwood, extract
1 oz. Magnesium, metal, ribbon
8 oz. Magnesium carbonate, powder
4 oz. Magnesium oxide
1 lb. Magnesium sulfate, cryst. (Epsom salts)
4 oz. Manganese chloride, cryst.
2 lb. Manganese dioxide, gran., free from carbon

4 oz. Manganese dioxide, powdered, c. p.
4 oz. Mercuric chloride, U. S. P., powder
8 oz. Mercuric oxide, red powder
4 oz. Mercurous nitrate, cryst.
4 oz. Mercury, metal, redistilled
10 g. Methyl orange
1 pt. Molasses, good quality
4 oz. Nickel (ous) sulfate, cryst.
1 pt. Olive oil
8 oz. Parchment paper
4 oz. Phenolphthalein solution (1%)
1 oz. Phosphorus, red, amorphous
4 oz. Phosphorus, yellow, sticks
4 oz. Picric acid, U. S. P., cryst.
4 oz. Potassium bitartrate (cream of tartar), c. p., powder
4 oz. Potassium bromide, U. S. P., cryst.
4 oz. Potassium carbonate, cryst., c. p.
1 lb. Potassium chlorate, small cryst., c. p.
4 oz. Potassium chloride, cryst., c. p.
4 oz. Potassium chromate, cryst., purified
1 lb. Potassium dichromate, cryst., technical
4 oz. Potassium ferricyanide, cryst., c. p.
4 oz. Potassium ferrocyanide, cryst., c. p.
1 lb. Potassium hydroxide, U. S. P., pellets
4 oz. Potassium iodide, cryst., c. p.
1 lb. Potassium nitrate, cryst., c. p.
4 oz. Potassium oxalate, cryst., c. p.
4 oz. Potassium permanganate, cryst., c. p.
8 oz. Potassium-sodium tartrate (Rochelle salt), U. S. P., powder
4 oz. Potassium sulfocyanate (thiocyanate), cryst., c. p.
1 oz. Pyrogallol
5 lb. Silica (sand)
4 oz. Silver nitrate, cryst., c. p.
1 lb. Soda lime, granular
4 oz. Sodium, metal
1 lb. Sodium acetate, powdered, anhydrous, c. p.
1 lb. Sodium bicarbonate (baking soda), U. S. P., powder
1 lb. Sodium bisulfite, dry, purified
4 oz. Sodium bromide, cryst., c. p.
1 lb. Sodium carbonate (washing soda), U. S. P., cryst.
8 oz. Sodium carbonate, dry, c. p.
5 lb. Sodium chloride (common salt), fine, white
2 lb. Sodium hydroxide, sticks, U. S. P.

4 oz. Sodium iodide, U. S. P.
1 lb. Sodium nitrate, cryst., c. p.
4 oz. Sodium peroxide, powder, purified
4 oz. Sodium phosphate (disodium), cryst., c. p.
1 pt. Sodium silicate solution (water glass), technical
1 lb. Sodium sulfate, cryst., c. p.
2 lb. Sodium thiosulfate (" hypo "), cryst., pea size
1 lb. Starch, corn
1 lb. Starch, potato
4 oz. Steel wool (# 00)
4 oz. Strontium chloride, cryst., c. p.
2 lb. Sugar, granulated
1 lb. Sulfur, roll
1 lb. Sulfur, flowers, sublimed
4 oz. Tin, mossy
4 oz. Tin, foil (0.0015" thick), pure
1 pt. Turpentine
2 lb. Zinc, mossy, technical
1 lb. Zinc, sheet
4 oz. Zinc oxide, powder
8 oz. Zinc sulfate, cryst., technical

MISCELLANEOUS SUPPLIES. It should be noted that this list does not include various materials which can readily be obtained at home or at the local stores, such as apples, baking powders, bread, butter, oleomargarine, soft and hard coal, calico, cotton cloth, woolen cloth and yarn, silk, linen, mixed goods, gasoline, gunpowder, iron nails, kerosene, machine oil, linseed oil, labels, matches, meat, cornmeal, oatmeal, milk, eggs, peanuts, pinks, Portland cement, rubber bands, Castile soap, vinegar, household ammonia, excelsior, wood chips and splints, candles, photographic developing powders, and yeast cakes.

DATE DUE SLIP

F. 255

QD 33 B62 1937 LAB-MAN-
BLACK NEWTON HENRY
NEW PRACTICAL CHEMISTRY

39891169 CURR HIST

000021691803

QD 33 B62 1937 lab.man.
Black, Newton Henry.
New practical chemistry :

39891169 CURR HIST

HISTORICAL
COLLECTION

Four-Figure Logarithms

N	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9
10	0000	0043	0086	0128	0170	0212	0253	0294	0334	0374	4	8	12	17	21	25	29	33	37
11	0414	0453	0492	0531	0569	0607	0645	0682	0719	0755	4	8	11	15	19	23	26	30	34
12	0792	0828	0864	0899	0934	0969	1004	1038	1072	1106	3	7	10	14	17	21	24	28	31
13	1139	1173	1206	1239	1271	1303	1335	1367	1399	1430	3	6	10	13	16	19	23	26	29
14	1461	1492	1523	1553	1584	1614	1644	1673	1703	1732	3	6	9	12	15	18	21	24	27
15	1761	1790	1818	1847	1875	1903	1931	1959	1987	2014	3	6	8	11	14	17	20	22	25
16	2041	2068	2095	2122	2148	2175	2201	2227	2253	2279	3	5	8	11	13	16	18	21	24
17	2304	2330	2355	2380	2405	2430	2455	2480	2504	2529	2	5	7	10	12	15	17	20	22
18	2553	2577	2601	2625	2648	2672	2695	2718	2742	2765	2	5	7	9	12	14	16	19	21
19	2788	2810	2833	2856	2878	2900	2923	2945	2967	2989	2	4	7	9	11	13	16	18	20
20	3010	3032	3054	3075	3096	3118	3139	3160	3181	3201	2	4	6	8	11	13	15	17	19
21	3222	3243	3263	3284	3304	3324	3345	3365	3385	3404	2	4	6	8	10	12	14	16	18
22	3424	3444	3464	3483	3502	3522	3541	3560	3579	3598	2	4	6	8	10	12	14	15	17
23	3617	3636	3655	3674	3692	3711	3729	3747	3766	3784	2	4	6	7	9	11	13	15	17
24	3802	3820	3838	3856	3874	3892	3909	3927	3945	3962	2	4	5	7	9	11	12	14	16
25	3979	3997	4014	4031	4048	4065	4082	4099	4116	4133	2	3	5	7	9	10	12	14	15
26	4150	4166	4183	4200	4216	4232	4249	4265	4281	4298	2	3	5	7	8	10	11	13	15
27	4314	4330	4346	4362	4378	4393	4409	4425	4440	4456	2	3	5	6	8	9	11	13	14
28	4472	4487	4502	4518	4533	4548	4564	4579	4594	4609	2	3	5	6	8	9	11	12	14
29	4624	4639	4654	4669	4683	4698	4713	4728	4742	4757	1	3	4	6	7	9	10	12	13
30	4771	4786	4800	4814	4829	4843	4857	4871	4886	4900	1	3	4	6	7	9	10	11	13
31	4914	4928	4942	4955	4969	4983	4997	5011	5024	5038	1	3	4	6	7	8	10	11	12
32	5051	5065	5079	5092	5105	5119	5132	5145	5159	5172	1	3	4	5	7	8	9	11	12
33	5185	5198	5211	5224	5237	5250	5263	5276	5289	5302	1	3	4	5	6	8	9	10	12
34	5315	5328	5340	5353	5366	5378	5391	5403	5416	5428	1	3	4	5	6	8	9	10	11
35	5441	5453	5465	5478	5490	5502	5514	5527	5539	5551	1	2	4	5	6	7	9	10	11
36	5563	5575	5587	5599	5611	5623	5635	5647	5658	5670	1	2	4	5	6	7	8	10	11
37	5682	5694	5705	5717	5729	5740	5752	5763	5775	5786	1	2	3	5	6	7	8	9	10
38	5798	5809	5821	5832	5843	5855	5866	5877	5888	5899	1	2	3	5	6	7	8	9	10
39	5911	5922	5933	5944	5955	5966	5977	5988	5999	6010	1	2	3	4	5	7	8	9	10
40	6021	6031	6042	6053	6064	6075	6085	6096	6107	6117	1	2	3	4	5	6	8	9	10
41	6128	6138	6149	6160	6170	6180	6191	6201	6212	6222	1	2	3	4	5	6	7	8	9
42	6232	6243	6253	6263	6274	6284	6294	6304	6314	6325	1	2	3	4	5	6	7	8	9
43	6335	6345	6355	6365	6375	6385	6395	6405	6415	6425	1	2	3	4	5	6	7	8	9
44	6435	6444	6454	6464	6474	6484	6493	6503	6513	6522	1	2	3	4	5	6	7	8	9
45	6532	6542	6551	6561	6571	6580	6590	6599	6609	6618	1	2	3	4	5	6	7	8	9
46	6628	6637	6646	6656	6665	6675	6684	6693	6702	6712	1	2	3	4	5	6	7	7	8
47	6721	6730	6739	6749	6758	6767	6776	6785	6794	6803	1	2	3	4	5	5	6	7	8
48	6812	6821	6830	6839	6848	6857	6866	6875	6884	6893	1	2	3	4	4	5	6	7	8
49	6902	6911	6920	6928	6937	6946	6955	6964	6972	6981	1	2	3	4	4	5	6	7	8
50	6990	6998	7007	7016	7024	7033	7042	7050	7059	7067	1	2	3	3	4	5	6	7	8
51	7076	7084	7093	7101	7110	7118	7126	7135	7143	7152	1	2	3	3	4	5	6	7	8
52	7160	7168	7177	7185	7193	7202	7210	7218	7226	7235	1	2	2	3	4	5	6	7	7
53	7243	7251	7259	7267	7275	7284	7292	7300	7308	7316	1	2	2	3	4	5	6	6	7
54	7324	7332	7340	7348	7356	7364	7372	7380	7388	7396	1	2	2	3	4	5	6	6	7
	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9

Four-Figure Logarithms — *Continued*

N	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9
55	7404	7412	7419	7427	7435	7443	7451	7459	7466	7474	1	2	2	3	4	5	5	6	7
56	7482	7490	7497	7505	7513	7520	7528	7536	7543	7551	1	2	2	3	4	5	5	6	7
57	7559	7566	7574	7582	7589	7597	7604	7612	7619	7627	1	2	2	3	4	5	5	6	7
58	7634	7642	7649	7657	7664	7672	7679	7686	7694	7701	1	1	2	3	4	4	5	6	7
59	7709	7716	7723	7731	7738	7745	7752	7760	7767	7774	1	1	2	3	4	4	5	6	7
60	7782	7789	7796	7803	7810	7818	7825	7832	7839	7846	1	1	2	3	4	4	5	6	6
61	7853	7860	7868	7875	7882	7889	7896	7903	7910	7917	1	1	2	3	4	4	5	6	6
62	7924	7931	7938	7945	7952	7959	7966	7973	7980	7987	1	1	2	3	3	4	5	6	6
63	7993	8000	8007	8014	8021	8028	8035	8041	8048	8055	1	1	2	3	3	4	5	5	6
64	8062	8069	8075	8082	8089	8096	8102	8109	8116	8122	1	1	2	3	3	4	5	5	6
65	8129	8136	8142	8149	8156	8162	8169	8176	8182	8189	1	1	2	3	3	4	5	5	6
66	8195	8202	8209	8215	8222	8228	8235	8241	8248	8254	1	1	2	3	3	4	5	5	6
67	8261	8267	8274	8280	8287	8293	8299	8306	8312	8319	1	1	2	3	3	4	5	5	6
68	8325	8331	8338	8344	8351	8357	8363	8370	8376	8382	1	1	2	3	3	4	4	5	6
69	8388	8395	8401	8407	8414	8420	8426	8432	8439	8445	1	1	2	2	3	4	4	5	6
70	8451	8457	8463	8470	8476	8482	8488	8494	8500	8506	1	1	2	2	3	4	4	5	6
71	8513	8519	8525	8531	8537	8543	8549	8555	8561	8567	1	1	2	2	3	4	4	5	5
72	8573	8579	8585	8591	8597	8603	8609	8615	8621	8627	1	1	2	2	3	4	4	5	5
73	8633	8639	8645	8651	8657	8663	8669	8675	8681	8686	1	1	2	2	3	4	4	5	5
74	8692	8698	8704	8710	8716	8722	8727	8733	8739	8745	1	1	2	2	3	4	4	5	5
75	8751	8756	8762	8768	8774	8779	8785	8791	8797	8802	1	1	2	2	3	3	4	5	5
76	8808	8814	8820	8825	8831	8837	8842	8848	8854	8859	1	1	2	2	3	3	4	5	5
77	8865	8871	8876	8882	8887	8893	8899	8904	8910	8915	1	1	2	2	3	3	4	4	5
78	8921	8927	8932	8938	8943	8949	8954	8960	8965	8971	1	1	2	2	3	3	4	4	5
79	8976	8982	8987	8993	8998	9004	9009	9015	9020	9025	1	1	2	2	3	3	4	4	5
80	9031	9036	9042	9047	9053	9058	9063	9069	9074	9079	1	1	2	2	3	3	4	4	5
81	9085	9090	9096	9101	9106	9112	9117	9122	9128	9133	1	1	2	2	3	3	4	4	5
82	9138	9143	9149	9154	9159	9165	9170	9175	9180	9186	1	1	2	2	3	3	4	4	5
83	9191	9196	9201	9206	9212	9217	9222	9227	9232	9238	1	1	2	2	3	3	4	4	5
84	9243	9248	9253	9258	9263	9269	9274	9279	9284	9289	1	1	2	2	3	3	4	4	5
85	9294	9299	9304	9309	9315	9320	9325	9330	9335	9340	1	1	2	2	3	3	4	4	5
86	9345	9350	9355	9360	9365	9370	9375	9380	9385	9390	1	1	2	2	3	3	4	4	5
87	9395	9400	9405	9410	9415	9420	9425	9430	9435	9440	0	1	1	2	2	3	3	4	4
88	9445	9450	9455	9460	9465	9469	9474	9479	9484	9489	0	1	1	2	2	3	3	4	4
89	9494	9499	9504	9509	9513	9518	9523	9528	9533	9538	0	1	1	2	2	3	3	4	4
90	9542	9547	9552	9557	9562	9566	9571	9576	9581	9586	0	1	1	2	2	3	3	4	4
91	9590	9595	9600	9605	9609	9614	9619	9624	9628	9633	0	1	1	2	2	3	3	4	4
92	9638	9643	9647	9652	9657	9661	9666	9671	9675	9680	0	1	1	2	2	3	3	4	4
93	9685	9689	9694	9699	9703	9708	9713	9717	9722	9727	0	1	1	2	2	3	3	4	4
94	9731	9736	9741	9745	9750	9754	9759	9763	9768	9773	0	1	1	2	2	3	3	4	4
95	9777	9782	9786	9791	9795	9800	9805	9809	9814	9818	0	1	1	2	2	3	3	4	4
96	9823	9827	9832	9836	9841	9845	9850	9854	9859	9863	0	1	1	2	2	3	3	4	4
97	9868	9872	9877	9881	9886	9890	9894	9899	9903	9908	0	1	1	2	2	3	3	4	4
98	9912	9917	9921	9926	9930	9934	9939	9943	9948	9952	0	1	1	2	2	3	3	4	4
99	9956	9961	9965	9969	9974	9978	9983	9987	9991	9996	0	1	1	2	2	3	3	3	4
	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9